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Some Observations on the Evaporation of Water from Cellulose*

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Abstract

The influence of water on the deterioration of cellulose is discussed, with special reference to deterioration at the brown line which forms when water rises in cotton by capillary action. The brown, fluorescent, water-soluble material results from the evaporation of water at the cellulose-water-air interface. Oxycellulose of the mildly reducing acidic type is formed. The oxidation appears to occur in part at the primary hydroxyl group, with the formation of polyglucuronic acid followed by chain scission and formation of soluble, low-molecular-weight fractions. The deterioration proceeds even at moderate temperatures and in diffused light indoors. The brown-line effect undoubtedly contributes significantly to the degradation of cotton during its production, processing, and use.

ONE of the aspects of a program of research on the chemical and photochemical degradation of cellulose initiated at the National Bureau of Standards in 1946 by the Office of The Quartermaster General included a study of the role of moisture in influencing the deterioration of cotton textiles. The general plan envisioned the isolation of several of the factors comprising textile "weathering"—radiant energy, moisture, atmospheric gases—in order to consider their effects singly and in combination in a controlled manner and thereby to assess the nature and relative contribution of each factor concerned.

Moisture has been shown to promote the degradation of cellulose in a variety of situations. Studies on this subject include that of Launer and Wilson [13] on the irradiation of paper in the near ultraviolet region in the presence of water vapor, that of Dorée and Healey [8] on the importance of moisture in the ozone oxidation of cellulose, and that of Wiegerink [22] on the increased degradation of cotton in high-temperature drying at higher concentrations of moisture. Fynn, Sands, and Campbell [9] observed substantial increases in the cuprammonium fluidities of fabrics exposed out of doors to the air and rainfall but shielded from direct solar radiation.

An experiment was devised to determine whether the action of wetting and drying, analogous to condi-

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tions fabrics may experience out of doors, could, of itself, contribute significantly to the degradation of cotton and filter-paper cellulose. Specimens were aged in an oven at 80°C for 80 days, wetting five times each day with just sufficient distilled water to saturate the specimens but with insufficient water to cause dripping and leaching. Control specimens, which were not wetted, were run simultaneously in the same oven. The wetted specimens soon acquired a yellowish or brownish color, which was particularly marked at the extremities of the wetted area as a series of brown lines. The cuprammonium fluidity values [14] of the aged specimens, shown in Table I, indicate the pronounced effect of the wetting in increasing the fluidity of the cellulose.

Although these results demonstrate the positive contribution of wetting and drying to breakdown of the cellulose, the temperature used was higher than that encountered in use (but not higher than that used in processing) of cotton textiles. This type of high-temperature experiment was thought to be unsatisfactory for the present purposes since, in addition to the complicating factor of heat, the aged wet cloth was found to exhibit a definitely acid reaction; the heat aging of acid-treated cotton cloth is known to degrade it [7]. The appearance of the cloth in the foregoing experiments was suggestive of the work of Bone [2], who in 1934 reported an interesting effect caused by the room-temperature evaporation of water from a purified cotton cloth suspended as a wick with one end immersed in water. The water rose in the strip of fabric by capillary action, and at the interface between the wet and dry areas a brown line formed which exhibited pronounced fluorescence when viewed in ultraviolet light and which enhanced the absorption of a basic dye such as methylene blue.

TABLE I. EFFECT OF WETTING AND DRYING IN OVEN AGING *

Material	Cuprammonium fluidity: Heated with wetting (rhes)	Heated without wetting (rhes)	Not heated (rhes)
Osnaburg†	18.4	7.8	2.8
Bleached print cloth‡	21.3	12.4	9.3
Analytical filter paper	19.1	16.7	11.0

* Specimens were heated in an oven at 80°C for 80 days, wetting 5 times each day with sufficient distilled water to saturate the specimen but insufficient water to cause dripping. Controls were not wetted.

† Purified by the method of Corey and Gray [4].

‡ Starch free, extracted with ethanol.

Since wetting and drying take place continuously over a small area at the water line, further investigation of wetting and drying at room temperatures was undertaken. Bone's results suggest that modification of the cellulose occurs under these mild conditions.

Recently, Bone and Turner [3] gave a fuller account of the experimental results of their "brown-line" investigations. In view of the unusual nature of this phenomenon and the suspension of work in this field at the National Bureau of Standards, it was considered desirable to publish our data. Many of the results of Bone and Turner have been confirmed, and additional observations concerning the nature of the material formed are reported herein. The general type of experiment involving the immersion of one end of a strip of fabric in water, as given by Bone, will be referred to hereinafter as a "brown-line" experiment.

General Observations

In general, several hours of running in a brown-line experiment were sufficient to produce a distinct yellow-brownish color at the wet-dry interface. The brown line could be produced in viscose rayon, filter paper, scoured cotton, and bleached cotton.

The rate of production of the brown material, which is readily water-soluble, was found to be surprisingly constant; a commercially bleached, sizing-free, alcohol-extracted print cloth was found to produce about 2 mg. per day per linear ft. of interface. This rate was the same when the experiments were run in the dark, in the diffuse daylight of the laboratory, and in the light from a nearby tungsten incandescent lamp.

At one point, attempts were made to estimate the quantity of fluorescent material that could be extracted from fabrics from brown-line experiments, using the Coleman Photofluorimeter and quinine sulfate solutions as standards. It was observed that brown fluorescent material could be extracted from fabrics subjected to a variety of degradative processes. Cotton cloth weathered out of doors for 6 months, or heated in the dark for 10 days, or exposed to the radiation from a carbon-arc source for 6 hrs. produced significant amounts of a brown fluorescent substance similar in appearance to that found in interface experiments. It was also found that the weight of extractable brown substance from brown-line experiments was increased about fourfold when

the fabric was exposed to radiation from a Westinghouse WL-794 Sterilamp placed about 6 in. from the fabric. Increasing the rate of air flow past the specimen served to shift the position of the interface by increasing the rate of evaporation, but the amount of extractable brown material did not increase. A sample of cloth that alternately was exposed in brown-line runs and water-extracted many times over a period of 8 months did not decrease in rate of formation of the extractable brown substance. This argues very strongly against the pre-existence of this substance, with subsequent concentration at the interface.

Spectroscopic analyses ruled out the possible role of iron or other inorganic constituents in producing the brown color at the interface. The only unusual increases in inorganic components were increased sodium and boron contents in one analysis. This could be attributed to leaching action on the Pyrex glassware employed, but the presence of such materials would not account for the effects observed.

Modification of Cellulose in Brown-Line Experiments

Modification of the substrate cellulose occurs in the course of brown-line experiments, as shown by increased cuprammonium fluidity, increased copper number, increased methylene blue absorption in the brown-line region, and, under certain circumstances, loss of strength at the interface. A recent paper by Rosenthal and Brown [17] shows that when purified cotton linters are subjected to alternate wetting and drying cycles, the cotton suffers appreciable modification, as indicated by the reduction in intrinsic viscosity of solutions of the cellulose when nitrated.

In the data presented herein, comparison is made between the analytical values for samples cut from the interface region and for controls cut from areas at some distance from the brown-line or from unexposed material. The direction of the differences, rather than their magnitude, is considered important because the amount of unmodified cellulose in the interface depends upon the width of the cut piece.

The fabrics used included a lightweight airplane cloth, desized and alkali-boiled, substantially in accordance with the procedure of Corey and Gray [4], and a starch-free commercially bleached print cloth which was Soxhlet-extracted with 95% ethanol. Both fabrics were extracted with water until the extract contained less than 10 mg. of solids per 100 g. of fabric. The laboratory distilled water used had a solids content of about 0.04 mg. per l.

Typical cuprammonium fluidity values [14] are given in Table II for samples exposed in a number of ways. The results indicate a substantial loss in the degree of polymerization at the evaporating boundary, which is indicative of an enhanced degradative activity at the interface.

The copper numbers of specimens from a bleached print cloth run for 5 months in a brown-line experiment were obtained using the Hägglund-Bertrand method [7]. The results showed that the cellulose substrate at the brown line exhibited increased reducing power as compared with the control, the copper numbers being 0.28 and 0.09, respectively.

The increased reducing power of the fabric at the interface was also shown in that silver was deposited in this area upon reaction with Tollens ammoniacal silver oxide reagent [15].

The presence of oxycellulose was also indicated by the test of Krajcinovic and Krajcinovic [12], in which the material is reacted with a diamine (benzidine) and a dye is developed by coupling with α-naphthol or resorcinol. The color was always strongly developed at the interface. Latent lines, not visible under ordinary light but fluorescing under ultraviolet illumination, were also strongly dyed in this test.

That the oxycellulose is of the acidic type is shown by the increased absorption of methylene blue. Figure 1 shows a sample of cloth that was exposed at room temperature for several months in a brown-line experiment and rinsed until no evidence of the brown lines could be seen in normal light. The cloth was then dyed with methylene blue and photographed. The intense dyeing at the interface boundaries is evident. Results obtained by the method of Davidson

TABLE II. CUPRAMMONIUM FLUIDITY VALUES IN BROWN-LINE EXPERIMENTS

Material	Conditions of experiment	Cuprammonium fluidity: At interface (rhes)	Cuprammonium fluidity: Control (rhes)
Print cloth	1 month, room temp.	15.3	9.3
Bleached airplane cloth (A)	3 weeks, 70°±2°F	10.8	9.0
Bleached airplane cloth (B)	3 weeks, 70°±2°F	25.2	17.2
Analytical filter paper	2 weeks, room temp.	12.1	11.0

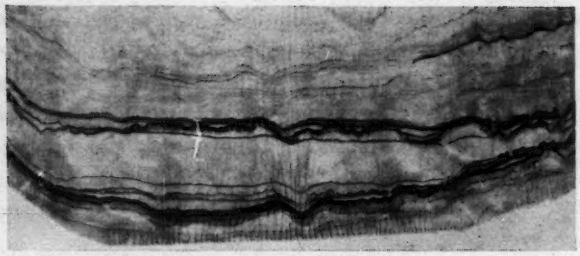

FIG. 1. *Methylene blue absorption of cloth from brown-line experiment after washing to remove the brown material.*

[5] at pH 8.1 and pH 2.7 are given in Table III. Measurements were made of horizontal sections across a fabric exposed in a brown-line run for 5 months. Cuprammonium fluidities of the same sections are shown for comparison. The increase in methylene blue absorption in the interface area is accompanied by a corresponding increase in the cuprammonium fluidity. The results indicate the increased carboxyl content of the modified cellulose in the interface area. Dorée [7] noted that for various oxycelluloses the ratio of methylene blue absorption in the neutral region to that at pH 2.7 is always greater than 2, whereas for hydrocelluloses produced by the action of acids on cellulose this ratio is less than 2, and frequently less than 1. By this criterion, therefore, it seems unlikely that the degradation is caused by concentration and drying of acid onto the fabric in the brown-line regions.

Runs were made using dilute acetic acid (pH 3.9) or ammonium hydroxide (pH 10.5) in place of water in brown-line experiments. After 30 days, methylene blue absorption at pH 8 at the washed interface area rose from 0.40 millimol per 100 g. to 0.55 and 0.69 millimol for the acid and alkaline solutions, respectively. The fact that the ammoniacal liquid appeared to be even more effective than the acid in increasing the dye uptake is further indication that concentration of acidic substances is not a cause for the observed deterioration of the fabric at the brown line.

Attention was given to the loss in breaking strength of the cloth in brown-line experiments. Such losses were found when the experiment was conducted at elevated temperatures for rather prolonged periods. In a typical experiment, a scoured Osnaburg was run in a brown-line experiment at 70°C for 368 hrs. After washing, yarns were removed, and the breaking strength was determined using a 10-in. gage length; the position of break was also determined and grouped with respect to the 1-in. class interval in which it occurred. Of 100 yarns, 40 broke at the interface area; this contrasts with the 10 per 100 expected to break in this region due to chance alone. The breaking load averaged 15% lower than a control tested simultaneously. In some experiments a strength loss of this order was not observed. The difficulty in obtaining consistent results seemed to be due to the difficulty in maintaining the interface area within narrow limits. Ordinarily, when the brown-line experiment was carried out at room temperature no loss in strength was found, but when the sample was heated subsequently a loss was found.

Nature of the Brown-Line Substance

As already noted, the colored material was found to be readily soluble in water, although heating the fabric tended to insolubilize a major portion of it. Dialysis of the water extract through cellophane membranes proceeded fairly rapidly. This observation indicates that no high-molecular-weight substances were present.

The freshly prepared water extract of the interface material was acid (pH of 4 to 4.5); after one month or more the extract approached neutrality. That this effect was not due to the presence of a volatile acid in the original substance was demon-

TABLE III. METHYLENE BLUE ABSORPTION AND CUPRAMMONIUM FLUIDITY IN BROWN-LINE EXPERIMENT

Section of fabric	Methylene blue absorption At pH 8.1 (millimols/100 g.)	Methylene blue absorption At pH 2.7 (millimols/100 g.)	Cuprammonium fluidity (rhes)
Topmost area*	0.52	0.10	15.2
Main interface region	.58	.18	18.5
Midway between interface and water	.40	.10	15.3
Just above water reservoir	.39	.09	9.7
Control	.38	—	7.3

* The interface occasionally rose to this area during humid periods.

strated by the lack of change in pH on evaporation to dryness and redissolving.

A number of the general classification tests described by Shriner and Fuson [19] were used to detect the presence of specific functional groups. Nitrogen, sulfur, and halogens were absent. The presence of unsaturated compounds was excluded by the negative reaction with bromine in CCl_4. The material gave a positive test with a potassium permanganate solution, indicating the presence of readily oxidizable compounds such as primary and secondary alcohols and aldehydes. However, the absence of aldehyde components was shown by the inability to form a bisulfite addition compound, an osazone, or a 2,4-dinitrophenylhydrazone. A positive test was obtained with Tollens reagent (ammoniacal silver oxide), which, in the absence of aldehyde, suggests a very reactive hydroxyl group. A positive reaction with periodic acid indicated the presence of 1,2-glycols or possibly of α-hydroxy acids. Oxidation with iodic acid was negligible, which left as possibilities, therefore, a number of polyhydric alcohols, hydroxy acids, glucose and glucose derivatives, among other compounds listed by Williams and Woods [23].

In view of the underlying probability that the brown-line material was derived from cellulose, a number of specific tests were made. A variety of tests for pentoses and hexoses gave only weakly positive results. The aniline acetate test for furfural [18] was negative on the extract and positive after hydrolysis of the brown residue with mineral acid. Since mineral acids are known to yield furfural from hydrolysis of uronic acids, a number of tests for glucuronic acid were made. These included the LeFevre-Tollens test using orcinol or phloroglucinol [15], the Denigues test with codeine [15], the Goldschmidt test using α-naphthol [15], and the naphthoresorcinol test of Tollens [15]. They gave positive results. A number of them, while widely used for the purpose, are known not to be specific for glucuronic acid. The Dische carbazole reaction, stated to be highly specific for hexuronic acids [6], gave a positive test. A cinchonine salt was prepared, giving a melting point of 204°C, which is identical with that given by Kalb and von Falkenhausen [11] for the cinchonine derivative of glucuronic acid.

Determinations of the uronic acid content of the interface area of a fabric sample were attempted using the method of Whistler, Martin, and Harris [21]. The rate of CO_2 evolution was notably higher than with a control, and followed an exponential rather than a linear function; this further indicated the presence of uronic acid material. Runs made with purified cellulose and added known quantities of glucuronic acid indicated a relatively low rate of CO_2 evolution; this made the quantitative determination of the uronic acid content of the interface area difficult in view of the small amounts available. It was estimated that less than 10% of the residue is uronic acid in character. Ruthenium red tests [1] used for the identification of pectic substances gave negligible staining, which showed that the purification procedure used effectively removed pectic material. The concentration of the latter, therefore, cannot be used to explain the apparent increase in uronic acid in the interface region.

Distillation under reduced pressure at 35°C yielded a distillate which gave positive reactions for formaldehyde (identified by its phenylhydrazone) and for formic acid [10]. The amounts of these substances, however, were estimated to comprise less than 1% of the brown extract.

Microscopic Observations

There was no evidence for unusual microbiological activity, as judged from microscopic examination of the brown-line region. The addition of thymol or of mercuric chloride to the water did not hinder the production of the usual effects, nor did the use of a 50% ethanol solution.

A water extract of the brown-line material from an experiment run for 3 months using extracted bleached print cloth was filtered, evaporated under reduced pressure to a small volume, and filtered again through a sintered-glass filter. The solution was permitted to stand in order to allow the remaining water to evaporate. The major portion of the residue consisted of very small colorless crystals embedded in a matrix of a light amber pasty substance.

An experiment was conducted in which a fabric run in a brown-line experiment was inoculated with *Aspergillus niger* and incubated at 30°C. The organism grew rapidly *along the brown line;* only after 4–5 days did the growth spread to other areas of the fabric.

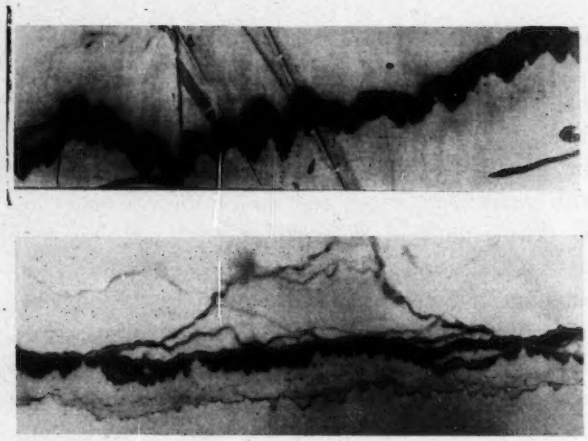

FIG. 2. Top—*Brown-line region on window curtain subject to frequent wetting by rain.* Bottom—*Brown-line region of purified airplane cloth from laboratory experiment.*

Discussion

The observations presented herein are in general agreement with those of Bone and Turner [3]. The data appear to lead to the conclusion that cellulose may be modified by the continuous evaporation of water at a cellulose-water-air interface under surprisingly mild conditions, with the production of oxycellulose of the mildly reducing acidic type. It is also suggested that a portion of the oxidation occurs at the primary hydroxyl group, with the formation of polyglucuronic acid followed by chain scission and the formation of soluble low-molecular-weight fractions. A recent study [16] has shown that by rigorous exclusion of oxygen, modification of the fabric in brown-line experiments can be prevented. This is good evidence of the oxidative nature of the reaction.

The work of Strachan [20], who recovered small amounts of soluble reducing substance by repeated extractions (and drying) of pure cellulose, may well be explainable by a phenomenon similar to the brown-line effect. In fact, there is a host of phenomena—the appearance of brownish areas on goods during shut-downs in paper mills and in textile finishing plants, unexplained "spots" observed in paper chromatography, to note but a few—which may be related to this effect. Figure 2 is illustrative of the possible importance of the brown-line effect in the home. The top photograph is of the lower portion of a window curtain which was wetted by rain from time to time over a period of 3 years. The brown line is similar to that obtained on cotton cloth in the laboratory over a period of 3 months; this is shown for comparison in the botton photograph of Figure 2. The curtain fabric was very tender and tore very easily in the interface region when pulled with the fingers.

The transport of material that was already present in small amounts on the samples or occluded within the fiber in this type of experiment undoubtedly confounded some of the observations made in the course of this work and is difficult to avoid. It is believed that the contribution of this effect is minor, although the migration of small amounts of modified substance that is already present may be of technological importance, as in some of the examples noted above.

It is not intended to discuss the various possibilities for explaining the mechanism of the brown-line phenomenon in view of the excellent discussion by Bone and Turner [3]. Further attempts at explanation would be highly speculative at this point. What is required is further experimentation using more rigorously defined celluloses of known history and type, with closer control of all of the ambient environment, and on a sufficient scale to produce larger amounts of material for study.

Literature Cited

1. Anderson, D. B., and Kerr, T., *Ind. Eng. Chem.* **30**, 48 (1938).
2. Bone, W. A., *J. Soc. Dyers Colourists* **50**, 307 (1934).
3. Bone, W. A., and Turner, H. A., *J. Soc. Dyers Colourists* **66**, 315 (1950).
4. Corey, A. B., and Gray, H. L., *Ind. Eng. Chem.* **16**, 853, 1130 (1924).
5. Davidson, G. F., *Shirley Inst. Memoirs* **21**, 47 (1947).
6. Dische, Z., *J. Biol. Chem.* **167**, 189 (1947).
7. Dorée, C., "The Methods of Cellulose Chemistry," 2nd edition, New York, D. Van Nostrand Co., 1947.
8. Dorée, C., and Healey, A. C., *J. Textile Inst.* **29**, T27 (1938).
9. Fynn, P. J., Sands, J. E., and Campbell, K. S., TEXTILE RESEARCH JOURNAL **18**, 350 (1948).
10. Grant, W. M., *Anal. Chem.* **20**, 267 (1948).
11. Kalb, L., and von Falkenhausen, F., *Ber.* **60B**, 2514 (1927).
12. Krajcinovic, M., and Krajcinovic, M., *J. Textile Inst.* **38**, T11 (1947).
13. Launer, H. F., and Wilson, W. K., *J. Research Natl. Bur. Standards* **30**, 55 (1943); RP1517.
14. Mease, R. T., *J. Research Natl. Bur. Standards* **22**, 271 (1939); **27**, 551 (1941).

15. Merck Index, 5th edition, Rahway, New Jersey, Merck and Company, 1940.
16. Preston, J. M., private communication.
17. Rosenthal, A., and Brown, R. K., *Pulp & Paper Mag. Can.* **51**, 99 (1950).
18. Scott, W. W., "Standard Methods of Chemical Analysis," 5th edition, New York, D. Van Nostrand Co., 1939.
19. Shriner, R. L., and Fuson, R. C., "The Systematic Identification of Organic Compounds," 2nd edition, New York, John Wiley & Sons, 1947.
20. Strachan, *Proc. Tech. Sect., Paper Makers' Assoc. Gt. Brit. & Ireland* **19**, 171 (1938).
21. Whistler, R. L., Martin, A. R., and Harris, M., *J. Research Natl. Bur. Standards* **24**, 13 (1940); RP1268.
22. Wiegerink, *J. Research Natl. Bur. Standards* **25**, 435 (1940).
23. Williams, R. J., and Woods, M. A., *J. Am. Chem. Soc.* **59**, 1408 (1937).

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The Oxidation of Cellulose by Ozone in Small Concentrations*

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Abstract

Laboratory experiments show that ozone in amounts which may occur in air at the earth's surface (up to 0.06 part per million) deteriorates cotton textiles when they are wet. The deterioration is slight in comparison with that from other elements of "weathering," such as light, heat, wetting and drying, and microorganisms. Fifty days of exposure to air containing 0.02 to 0.06 part of ozone per million was required to reduce the breaking strength of wet print cloth 20% and increase the fluidity in cuprammonium solution from 8 to 16 rhes. When exposed dry, the cloth underwent little or no change in these characteristics. An increase in concentration of ozone led to an increase in the deterioration of the cellulose.

OZONE is present in the atmosphere at the earth's surface in amounts varying from 0 to 0.06 part per million of air [2], being formed from oxygen by short wave-length ultraviolet radiation in the upper atmosphere. Until recently, the importance of ozone in weathering phenomena had not been realized, probably because it was not expected that a substance present in such minute amounts could have any significant effect. However, the recent work of Crabtree and Kemp [2] and Newton [9] shows that serious deterioration of strained rubber occurs upon outdoor exposure through the action of atmospheric ozone. In view of this, a study concerned with the possible role of ozone in the deterioration of cotton textiles exposed to the weather was initiated at the National Bureau of Standards in 1946 by the Office of The Quartermaster General. This investigation was part of a program aimed at segregating some of the elements comprising "weathering"—radiation, moisture, atmospheric gases—in order to assess the potential role of each.

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Dorée and coworkers [3, 4, 5] studied the oxidation of cellulose by ozone, using concentrations of the order of 2 g. of ozone per 100 g. of gas, and reported that under moist conditions oxycelluloses of high reducing power are produced. In the experiments described herein, cotton cloth was exposed for fairly long periods to lower concentrations of ozone, including amounts corresponding to the ozone content of the atmosphere.

Methods

A schematic diagram of the apparatus is shown in Figure 1. The ozone generator used in most of the runs was a WL-794 Westinghouse Sterilamp, which, after an initial breaking-in period of a day or two, proved to be an excellent stable source for the low ozone production required. In some of the preliminary runs, a silent electric-discharge ozone generator was used.

Ozone concentration was determined by bypassing the exposure chamber periodically and absorbing the ozone in a buffered potassium iodide solution kept in a light-tight box, essentially as described by Crabtree and Kemp [1]. It was found more convenient, however, to estimate the iodine liberated spectrophotometrically using a Beckman Spectrophotometer than by the electrometric method of Crabtree and Kemp. At 355 mμ, 1.0×10^{-5} g. of iodine per ml. could be determined to within 2%. Based on the periodic analyses, minor adjustments in the flow rates could be made to compensate for any changes in the system.

The course of the reaction of the ozone with the cellulose was followed by the change in cuprammonium fluidity, using the method of Mease [8], and the change in breaking strength of the cloth.

Results and Discussion

A preliminary run was made using scoured cotton Osnaburg and a silent electric-discharge ozonizer. When this source was used, the ozone content of the air was rather variable, ranging between 100 and 230 p.p.m., and averaging 160 p.p.m. When the fabric was kept moist, substantial degradation of the cellulose was observed, as shown in Table I.

Decided bleaching of the moist fabric occurred in the first 60 hrs.; little further bleaching was found with further exposure. An indication of the increased brightness of the bleached fabric is given by the rise

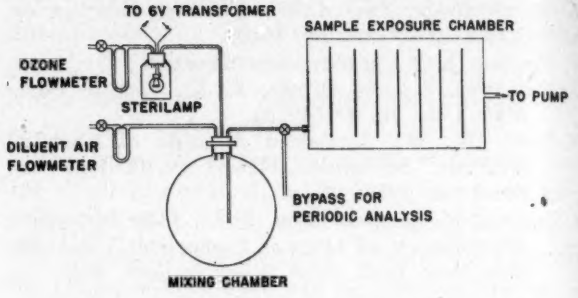

FIG. 1. *Schematic drawing of apparatus.*

in reflectance from 54% to 66%, as measured with a Hunter Reflectometer using the blue filter. An air-dry sample which was not moistened showed little fluidity change in 60 hrs. of exposure under these conditions and was not perceptibly whitened.

Apparatus using the Westinghouse Sterilamp was set up and adjusted to give ozonized air containing 1.0 p.p.m. of ozone. This corresponds to less than twenty times the maximum usually found in the atmosphere. The extent of the cellulose degradation under these conditions is indicated in Table II, the fabric being water-saturated continuously, except as noted in the table. The results indicate that substantial modification of moist cotton may occur with less than twenty times the atmospheric concentration of ozone.

A run was made using ozonized air of ozone content ranging between 0.02 and 0.06 p.p.m. The fabric was a scoured cotton duck, water-saturated once a week, with moisture added to the air by bubbling the air through water so that the moisture content of the cloth was never less than 50%. The rate of deterioration, as shown by cuprammonium fluidity values, is given in Table III. The specimen at the end of the 1200-hr. (or 50-day) exposure period showed a loss in breaking strength of 20%. Similar results were obtained with a commercially bleached cotton

TABLE I. CUPRAMMONIUM FLUIDITY OF COTTON OSNABURG EXPOSED WHILE MOIST TO OZONIZED AIR CONTAINING 100 TO 230 P.P.M. (AVERAGE 160 P.P.M.) OF OZONE

Duration of exposure (hrs.)	Cuprammonium fluidity (rhes)
0	2.7
60	26.4
108	38.2
143	41.5

TABLE II. CUPRAMMONIUM FLUIDITY OF COTTON OSNABURG EXPOSED WHILE MOIST TO OZONIZED AIR CONTAINING 1.0 P.P.M. OF OZONE

Duration of exposure (hrs.)	Cuprammonium fluidity (rhes)
0	2.8
42	3.4
67	4.5
213	5.0
432	5.9
529*	10.2
720*	12.4

* Sample was water-saturated once daily rather than continuously in the last 300 hrs.

print cloth that was similarly exposed except that one end of the cloth was immersed in water and the entire surface was kept wet continually by capillary flow. The data are also shown in Table III. A wet print cloth control that was not exposed to the ozone had a cuprammonium fluidity of 9.0 rhes at the end of 1500 hrs., indicating that virtually all of the degradation undergone by the fabric is attributable to the action of this small concentration of ozone.

Conclusions

It is concluded that ozone at very low concentrations can oxidize wet cellulose. Since dry cotton was substantially unaffected in these experiments, it is possible that the mode of operation of the ozone is primarily through solution in the water, ozone being considerably more soluble than air at room temperature. Henry's Law constants for the solubilities given by "International Critical Tables" [6] are of the order of 10^6 and 10^7 for ozone and air, respectively, but it is difficult to estimate the ozone content of the liquid on the fiber from the present data since the concentration would depend upon the equilibrium between solubility, decomposition, and amount reacted.

With respect to degradation in weathering, it is seen that 50 to 60 days of exposure under conditions that are ideal for ozone attack are required to produce appreciable modification (*i.e.*, 20% strength loss, and a fluidity rise from 2.6 to 9.5 rhes) of cellulose in the form of cotton duck. Under field conditions, in most climates the requisite circumstances of high ozone content of the atmosphere and sample wetness could hardly be expected to exist more than 10% of the time. Therefore, an estimated 500 or 600 days might be required to reach this stage of degradation in, for example, tentage, due to the action of ozone alone in natural weathering. Since unprotected fabrics exposed for such periods have generally reached a considerably more advanced state of degradation [7], it is concluded that the contribution of atmospheric ozone to weather deterioration is relatively small if synergistic effects are not importantly involved.

TABLE III. CUPRAMMONIUM FLUIDITY OF COTTON CLOTH EXPOSED WHILE MOIST TO OZONIZED AIR CONTAINING 0.02 TO 0.06 P.P.M. OF OZONE

	Duration of exposure (hrs.)	Cuprammonium fluidity (rhes)
Duck	0	2.6
	200	2.8
	680	4.0
	960	6.8
	1200	9.5
Print cloth	0	8.2
	200	8.7
	510	9.4
	650	12.0
	865	12.7
	1500	16.5

Acknowledgment

The able assistance of Karl Plitt in carrying out the experiments is acknowledged with thanks.

Literature Cited

1. Crabtree, J., and Kemp, A. R., *Ind. Eng. Chem., Anal. Ed.* **18**, 769 (1946).
2. Crabtree, J., and Kemp, A. R., *Ind. Eng. Chem.* **38**, 278 (1946).
3. Cunningham, M., and Dorée, C., *J. Chem. Soc.* **101**, 497 (1912).
4. Dorée, C., *J. Chem. Soc.* **103**, 1347 (1913).
5. Dorée, C., and Healey, A. C., *J. Textile Inst.* **29**, T27 (1938).
6. "International Critical Tables," Vol. III, p. 257 (1928).
7. Little, R. W., "Flameproofing Textile Fabrics," New York, Reinhold Publishing Corp., 1947, chap. VII.
8. Mease, R. T., *J. Research Natl. Bur. Standards* **22**, 271 (1939); **27**, 551 (1941).
9. Newton, R. G., *Rubber Chem. and Technol.* **18**, 504 (1945).

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The Geometry of Plain and Rib Knit Cotton Fabrics and Its Relation to Shrinkage in Laundering

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Abstract

The geometry of six plain and six rib circular-knit cotton fabrics with three different finishes was studied. Data on both gray and finished fabrics for stitch length, diameter of yarn, and wale and course spacings were used in evaluating the equations derived by Peirce for length of yarn in one stitch and for weight per unit area. Measured stitch length agreed with that calculated from Peirce's formula, $l = 2p + w + 5.94d$, when a specific volume of 1.1 was assumed for the yarn. When the measured value for the yarn diameter was used, the stitch length was given by $l = 2p + w + 4.56d$. Weight of the fabrics calculated by Peirce's formula showed good agreement with that obtained by weighing a known area.

In laundering tests, it was found that the shrinkage of the yarn was negligible, usually less than 1% in the finished fabrics. Curves relating the wale and course spacings of the unlaundered gray fabrics conformed to no orderly pattern. The gray material shrank in length and stretched excessively in width. Wale spacings plotted *vs.* course spacings in the laundered gray and finished fabrics followed parabolic curves. The finished fabrics in which wale and course spacings approximated the parabolic relationship of the laundered fabrics changed the least in the length and width dimensions. These were the tighter-knit fabrics of two of the finishes. On the other hand, the fabrics having the greatest knitting stiffness shrank the most in area.

THE GEOMETRY of cloth structure has been given considerable attention by Peirce [3], Womersley [6], and Pollitt [5]. Woven fabrics have been analyzed by these workers in terms of geometrical quantities such as diameter of yarn, spacings of yarn in warp and filling, and crimp. These quantities have been measured and reported for many specific fabrics. Peirce's studies of the geometry of woven cloth before and after laundering have revealed the causes of shrinkage in terms of yarn shrinkage, swelling, and crimp redistribution.

Peirce is the only worker known to have attempted a study of the geometry of knitting [4]. Using diameter of yarn and wale and course spacings, he derived equations for determining the length of yarn in one stitch of flat plain knitted fabric and for weight per unit area of such cloth. However, experimental data from knit goods were not used to verify these theoretical relationships. The laboratory investigation reported here was undertaken to supply such data, and, through a study of the geometry of plain and rib knit cotton goods, to obtain basic information regarding the stretching and shrinking of these materials when laundered.

Materials

The fabrics studied were knit under controlled conditions in the Textile Research Department of the American Viscose Corporation. They were of medium-staple 30/1 combed Peeler cotton yarn having Z twist of 18 turns per in. in two types, flat plain and 1 × 1 rib. A 28-cut circular machine of 20-in. diameter was used for all the flat knit; and a 12-cut circular machine of 18-in. diameter was used for all the rib knit. Each type was knit with the following courses per inch: 24, 28, 32, 36, 40, and 44. The plain knit fabrics are identified by small letters (a to f), the rib knit by capital letters (A to F).

Some of the yardage of the gray goods was set aside, and the remainder was scoured and bleached. After these two processes the goods were divided into three lots and were given three different mechanical

finishing treatments. The first lot, designated "finish I," was calendered in the usual way. The second lot, designated "finish II," was run through wide spreaders to remove distortion which occurred in scouring and bleaching. The knit fabrics were relaxed during drying, and were then calendered. The third lot, designated "finish III," was dried on a loop dryer and finished on a tensionless folding calender.

The finished widths, irrespective of the three treatments, were nearly identical for each construction. The flat knit varied in width from 32 in. for the fabrics knit with 24 courses per in. to 26.5 in. for those of 44 courses per in. Similarly, the finished widths of the 1 × 1 rib knit varied between 17 and 13.5 in.

Method

Data on dimensional change in laundering and on fabric properties were obtained for each material. Three 15 in. × 15 in. specimens of each fabric were used for measuring dimensional change in laundering. Since there is no standard test method for measuring shrinkage in laundering for knit goods, the procedure used was the A.S.T.M. standard method for woven cloth [1]. After the last rinse the specimen, without distortion, was floated in water over a wire screen placed on the bottom of a tray. Both the fabric and screen were lifted from the tray. The fabric was not removed from the screen until dry.

Pertinent data regarding the yarn and fabrics were obtained by the procedures described below, and were applied to Peirce's equations to determine whether they followed the theoretical relationships which he derived.

(*a*) The yarn number was determined on a yarn numbering balance.

(*b*) The diameter of the yarn was found by using a camera lucida. A maximum and a minimum were measured on each of twenty specimens.

(*c*) The stitch length was determined by measuring, under a tension of 6 g., the length of yarn taken up in 300 loops raveled from the fabric. Forty measurements, twenty from each of two different places on the fabric, were made.

(*d*) The weight per sq. yd. was obtained by weighing 2-in. squares on a torsion balance calibrated in oz. per sq. yd. Five specimens were taken for the test.

(*e*) The wale and course counts were taken at ten different places on the fabric before and after laundering.

Results and Discussion

The formulas derived by Peirce for flat plain knitting are:

(*1*) for the length of yarn, l, in a "unit cell," or in one stitch:

$$l = 2p + w + 6 \cdot \sqrt{4/\pi} \cdot \sqrt{g/\rho},$$

which can also be expressed as

$$l = 2p + w + 5.94d;$$

(*2*) for the weight, W, per unit area of cloth:

$$W = \frac{l\ g}{w\ p},$$

where p is the course spacing, w the wale spacing or width per wale, g the weight per unit length of yarn, ρ the yarn density, and d the diameter of yarn.

The relationships in rib knitting can be deduced from the above as follows: The unit cell in 1 × 1 rib knit can be treated as having an area of 2 wp.* The number of wales per unit length can be taken as the total number of wales per unit length on both sides of the cloth, which has been knit by the two banks of needles. Thus, the same relationships for flat plain knitting may be applied to the rib knit if l is the length of one loop and not the length in a unit cell, and w is the reciprocal of the total number of wales per unit length on both sides of the cloth.

Length of Stitch

The data on length of stitch for the gray and finished knit fabrics are given in Table I; the relationship of stitch length to wale and course spacings is shown in Figure 1.

Before evaluating equation (1), the diameter of the yarn was determined by direct measurement and by calculations from the weight, using different densities. The weight of 1 m. of yarn (0.02029 g.) was calculated from the experimental yarn number (29.11, cotton system).

The values obtained for yarn diameter, d, were: 8.725 mils using camera lucida; 8.456 mils using yarn density 0.56; 6.634 mils using specific volume 1.1; 5.163 mils (critical diameter) using fiber density 1.50.

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* Peirce stated, "In the rib knit, there are twice as many loops per unit cell, enclosed within one course length and the repeat of the structure, as seen from one side" [4], p. 146).

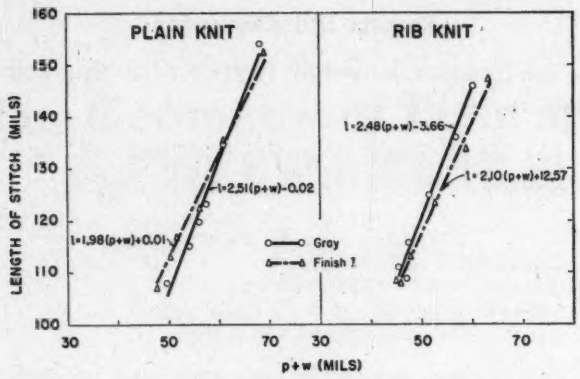

FIG. 1. *The relationship of stitch length to course and wale spacings,* p + w, *of gray and finished knit fabrics.*

The measured value (8.725 mils) is higher than the 6.634 mils value calculated from the specific volume of 1.1, which Peirce assumed for cotton yarn used in woven fabrics [3]. The measured value is also higher than that calculated from the initial cotton yarn density (0.56), which Barella used in his study of critical yarn diameter [2]. All of the values are considerably higher than the value known as "critical diameter."

The experimental data on stitch length for both plain and 1 × 1 rib knit fabrics followed the relationship

$$l = 2p + w + 5.98d$$

when a specific volume of 1.1 was assumed for the yarn in calculating yarn diameter. This equation is

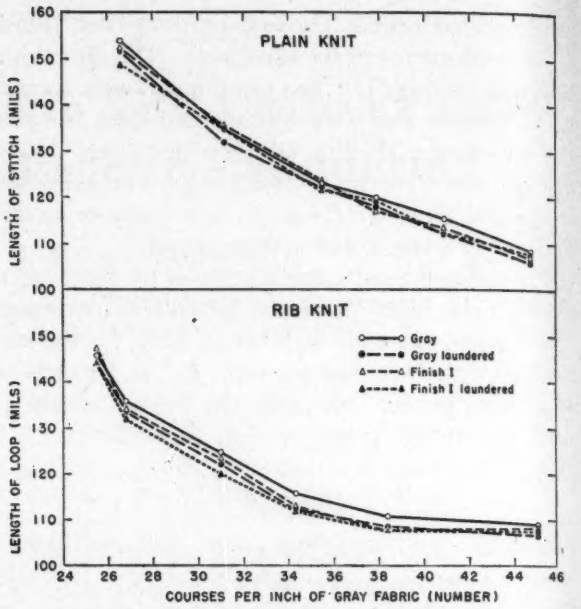

FIG. 2. *Stitch length of knit fabrics of various courses per inch before and after five launderings.*

in agreement with Peirce's equation, $l = 2p + w + 5.94d$, for stitch length.

In using the experimental value for yarn diameter ($d = 8.725$ mils), the data followed the relationship

$$l = 2p + w + 4.56d,$$

which gives a somewhat lower value for stitch length than does Peirce's equation.

TABLE I. WALE AND COURSE COUNT, STITCH LENGTH, AND WEIGHT OF KNIT COTTON FABRICS *

Fabrics	Knitted courses per in.†	Counted wales and courses per in. Gray Wales	Gray Courses	Finish I Wales	Finish I Courses	Length of stitch Gray (.001 in.)	Finish I (.001 in.)	Weight per sq. yd. Gray Calculated (oz.)	Gray Experimental (oz.)	Finish I Experimental (oz.)
Plain knit										
a	24	32.6	26.6	31.5	26.9	154.0	152.2	3.15	3.34	2.88
b	28	34.4	31.2	33.0	32.6	134.5	135.1	3.40	3.56	3.10
c	32	34.2	35.5	34.1	37.2	122.9	122.4	3.52	3.68	3.50
d	36	33.8	37.9	36.7	41.6	119.7	117.0	3.61	3.84	3.70
e	40	33.9	40.9	37.0	42.9	115.2	113.0	3.76	3.90	3.76
f	44	36.7	44.8	37.1	48.2	108.1	106.9	4.19	4.18	4.04
1×1 Rib knit										
A	24	47.6	25.5	44.4	24.5	145.8	147.4	4.17	4.50	3.40
B	28	51.1	26.8	44.0	27.7	135.9	134.0	4.38	4.54	3.54
C	32	51.9	31.0	47.8	31.6	124.8	123.4	4.73	4.70	4.14
D	36	54.6	34.3	49.5	36.3	115.7	112.9	5.10	5.22	4.42
E	40	51.3	38.4	53.3	37.0	110.8	108.1	5.14	5.26	4.76
F	44	40.5	45.1	50.5	39.2	108.8	108.3	4.68	4.84	4.78

* Data for properties of fabrics with finishes II and III were approximately the same as those for fabrics with finish I.
† Courses were counted with the gray fabric in a relaxed condition after removal from the knitting machine.

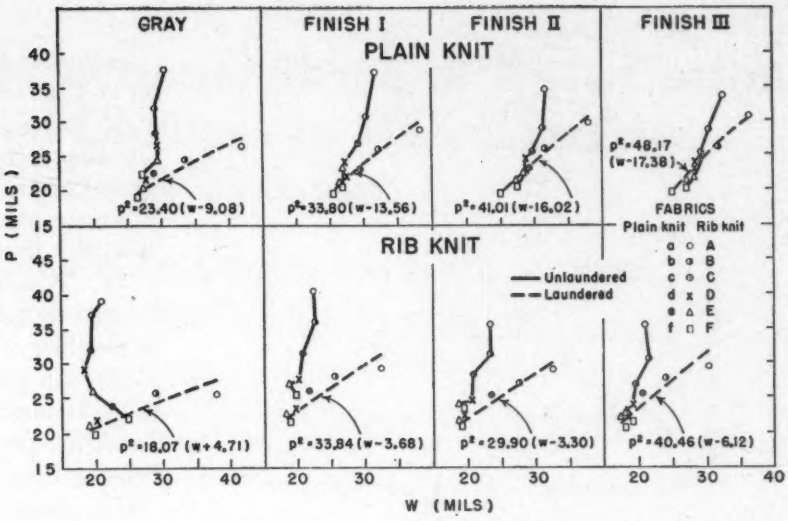

FIG. 3. *Wale* (w) *and course* (p) *spacings of knit fabrics before and after five launderings.*

Weight

For the calculations of weight of the knit fabrics, Peirce's formula was transformed to

$$W = \frac{24}{35}\frac{n_c n_w l}{N}$$

so that weight in oz. per sq. yd. could be found in terms of courses per in., n_c; wales per in., n_w; length of stitch in in., l; and yarn number in the cotton system, N. The calculated weights of the fabrics showed good agreement with those obtained by weighing a known area (see Table I).

Effect of Shrinkage in Laundering upon the Geometry of Knit Fabrics

Measurements of yarns raveled from all of the fabrics before and after laundering revealed that the shrinkage of the yarn was slight (see Figure 2). After five launderings, shrinkage in the stitch length of the plain and rib knit finished fabrics usually ran less than 1%, while in the gray goods it ranged from 1% to 3%.

On the other hand, marked changes in the relationship of wale and course spacings occurred during laundering. A study of the data for the laundered goods revealed that wale spacings plotted against course spacings follow parabolic curves. Therefore, the data were fitted to a quadratic equation using the general form for a parabola:

$$p^2 = a\ (w + b).$$

The relationship of the wale and course spacings for the unlaundered gray and for the unlaundered finish I of the plain knit fabric was quite different from that for these fabrics after laundering (see Figure 3). But for finishes II and III, the wale *vs.* course spacing curves for the unlaundered materials more nearly approached the parabolic curves for the laundered fabrics. The portion of the curves for the more loosely knit unlaundered fabrics departed the most from that of the laundered fabrics.

Likewise, the gray and finish I unlaundered rib fabrics were quite different from the laundered with respect to the relationship of wale and course spacings (see Figure 3).

The great extent to which finishing can diminish the shrinking and stretching of knit goods in laundering is shown in Figure 4. In both the plain and rib knit fabrics, the least dimensional change occurred in those so finished that the wale and course spacings followed a parabolic relationship.

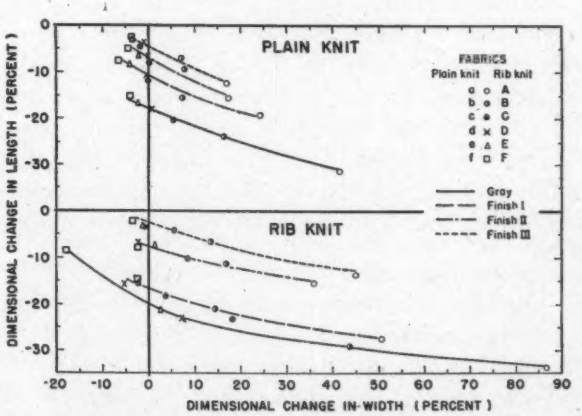

FIG. 4. *Dimensional change in length and width of knit fabrics in five launderings.*

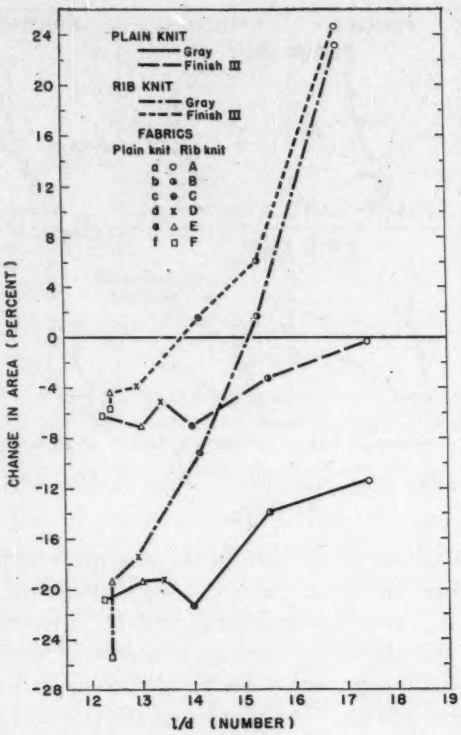

FIG. 5. *Relationship of (stitch length)/(diameter of yarn) to change in area of knit fabrics during five launderings. Diameter of yarn equals 8.725 mils.*

The ratio l/d is a convenient "cover factor" to use as a measure of tightness of knitting. In the plain knit fabrics l/d ranged from 17.0 to 12.3, and in the rib knit from 16.9 to 12.4. Peirce has stated that knitting becomes crammed when $l/d = 16$ [4]. According to this standard, all of the knitting was crammed except that for the most loosely knit fabrics of the plain (fabric a) and of the rib knit (fabric A).

Shrinkage in area was greater for the tightly knit fabrics than for those that were loosely knit. However, the relationship of l/d to change in area in laundering shows that knitting stiffness alone does not determine the magnitude of dimensional change in area (see Figure 5). For example, the more tightly knit gray fabrics exhibited excessive shrinkage, while those with finish III shrank only moderately.

Summary

In this study of the geometry of certain plain and rib knit cotton fabrics, it was found that:

(1) Measured stitch length agreed with that calculated from Peirce's formula, $l = 2p + w + 5.94d$, when specific volume of 1.1 was assumed for the yarn. When the measured value for the yarn diameter was used, the stitch length was given by $l = 2p + w + 4.56d$. Weight of the fabrics calculated by Peirce's formula showed good agreement with that obtained by weighing a known area.

(2) The yarn in the finished fabrics shrank a negligible amount in laundering. Therefore, the shrinkage of the yarn contributed little to the dimensional change of the fabric.

(3) Those finished fabrics in which the wale and course spacings before laundering followed a parabolic relationship similar to that of the laundered fabrics exhibited the least change in length and width.

(4) The fabrics having the greatest knitting stiffness shrank the most in area. In some of the knit fabrics the increase in width more than offset the shrinkage in length so that the area of the laundered fabric exceeded that of the original specimen.

Acknowledgment

Grateful acknowledgment is made to Harriet D. Harvey and Elinora Dent for laboratory assistance, and to Mary A. Fajen for assistance in preparation of the data reported in this paper.

Literature Cited

1. Am. Soc. Testing Materials, Committee D–13, "A.S.T.M. Standards on Textile Materials (with Related Information)," Philadelphia, 1950.
2. Barella, A., TEXTILE RESEARCH JOURNAL **20**, 249 (1950).
3. Peirce, F. T., *J. Textile Inst.* **28**, T45 (1937).
4. Peirce, F. T., TEXTILE RESEARCH JOURNAL **17**, 123 (1947).
5. Pollitt, J., *J. Textile Inst.* **40**, P11 (1949).
6. Womersley, J. R., *J. Textile Inst.* **28**, T97 (1937).

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Influence of Cross-Sectional Shape of Raw Cotton on Dimensional Changes in Mercerization Without Tension

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Abstract

The relation of the cross-sectional shape of the raw fiber to the changes that occur upon mercerization without tension was studied for a number of cotton varieties representing a wide range of maturities.

Perimeters and diameters of fiber cross sections were measured by the use of an especially designed instrument. Areas also were measured.

It was found that the percent increase in area and the percent decrease in perimeter caused by loose mercerization are dependent upon the cross-sectional shape of the original raw fibers. Flat fibers shrank more in perimeter and increased more in area than did round fibers. This was partly explained as being due to a change toward circularity.

THIS PAPER reports data, obtained during an investigation of the fundamental swelling properties of cotton, which show that the cross-sectional shape of the raw cotton fiber is related to the changes that occur in mercerization without tension.

The undried fibers from a fresh, undried boll of cotton, which are tubular in form, consist of a thin primary wall surrounding a secondary wall, the thickness of which depends upon the maturity of the fiber. The central opening, or lumen, ranges from very small for thick-walled (mature) fibers to a large proportion of the whole cross-sectional area for thin-walled (immature) fibers [5]. As the fiber dries, the lumen collapses. This gives the fiber cross section various shapes, ranging from almost circular for mature fibers to flat for immature fibers.

It has long been known that treatment of the raw cotton fiber in strong swelling agents, as in the mercerization process, causes the fiber to swell toward a circular cross-sectional shape by the thickening of the secondary wall. It has been shown that the primary wall tends to limit the swelling, and that the resulting pressure closes the lumen [7, 15]. This restricting action was attributed to the alignment of the fibrils of the primary wall, which are nearly perpendicular to the axis of the fiber [4]. The primary wall, however, may be ruptured by very strong swelling agents [9].

The relation of cross-sectional shape to swelling in water was studied by Skau [14] and Moore [12]. Skau predicted that, although thin-walled fibers might reach their maximum swelling in water, thick-walled fibers might be prevented from doing so by the constricting action of the primary wall and might develop a real swelling pressure. Observations by Moore *et al.* [12] indicate that the shape of the fiber cross sections is changed little with swelling in water. This led to the belief that the secondary wall offered considerable resistance to deformation by swelling.

Calvert and Summers [6] showed that upon washing out the mercerizing agent and drying, the fiber shrank in diameter, but retained the more or less circular cross-sectional shape assumed in the mercerizing solution. There was a decrease in both cross-sectional area and perimeter from the swollen stage; but, compared to the raw fiber, the area of the cross section was larger and the perimeter was smaller.

Marsh [11] reported a considerable decrease in length upon mercerization without tension, which he attributed to the alignment of the inner layers of fibrils which form a connected network aligned roughly parallel to the fiber axis. Swelling of the more amorphous cellulose between the fibrils would

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cause shortening of the network in the direction of alignment of the fibrils.

Experimental

Samples

In the present study, sixteen samples from nine varieties were used, more than one sample of some varieties representing different locations and different crop years. The varieties selected had the range of length, maturity, and fiber fineness shown by the values given in Table I.

Procedures

Mercerization.—Two bundles of approximately 250 fibers each were taken from the modal-length group of the length distribution. One bundle was retained as a control, and the other was mercerized in 18% sodium hydroxide solution at 70°F for 10 min., washed thoroughly, and dried in an oven at 100°C. No tension was applied during the process. In order to insure uniformity of treatment, the sixteen samples were processed simultaneously.

Cross-Sectioning and Measuring.—The cross-sectioning technique developed by Karrer and Bailey [10] was used to obtain photomicrographs of raw and mercerized samples of each cotton. The photographs were enlarged to a magnification of 1,000 diameters, and linear and area measurements were taken on 100 fibers in each photomicrograph. Each sample was sorted, mercerized, and measured twice, giving a total of 200 measurements for each sample.

The dashed lines in Figure 1 indicate the linear dimensions measured. The ribbon width, d_1, corresponding roughly to the minor axis of an ellipse, is the shortest width of the fiber. For very thin-walled fibers it is an average width. The ribbon thickness, d_2, corresponding to the major axis of an ellipse, is measured along the center line of the fiber cross section. P_R and P_M represent the perimeters of the primary wall of the raw and mercerized fibers, respectively.

These measurements were made with an instrument that was designed at the Southern Regional Research Laboratory to facilitate the method of taking linear measurements on irregular figures. This instrument (Figure 2) records the rotation of a motor-driven knurled wheel, A, as it pulls the photomicrograph beneath it at a rate of 30 cm. per min. The wheel is held in position by the drive shaft, K, and

TABLE I. SAMPLE IDENTIFICATION

Sample No.	Variety	Length at upper quartile* (in.)	Maturity† (% mature)	Fineness‡ (μg./in.)
1	Mexican Big Boll	1.16	94	5.35
2	Mexican Big Boll	1.15	89	5.25
3	Acala Rogers	1.14	90	3.85
4	Sea Island	1.68	86	2.40
5	Acala Rogers	1.19	92	4.40
6	Wilds 5	1.33	84	3.55
7	Wilds 5	1.31	85	3.80
8	Wilds 5	1.36	86	3.88
9	Half & Half	0.87	92	5.63
10	Half & Half	0.88	84	5.35
11	Mexican Big Boll	1.11	85	5.05
12	Acala 1517	1.31	77	3.33
13	Half & Half	0.87	87	5.38
14	Farm Relief	1.14	83	4.60
15	Delfos 4	1.23	80	4.10
16	Empire	1.18	78	3.98

* Determined from length distributions prepared by the A.S.T.M. method [2].

† Determined by the sodium hydroxide method [3].

‡ Determined with the Micronaire using the linear scale furnished on the instrument.

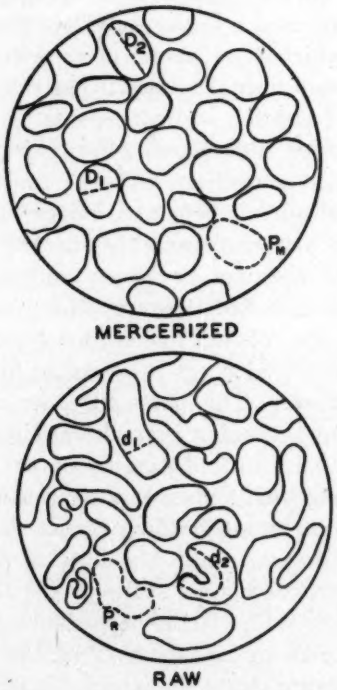

FIG. 1. *Outline of photomicrographs of cross sections of cotton fibers, showing linear dimensions measured:* d_1, *minor axis of raw fiber;* d_2, *major axis of raw fiber;* P_R, *perimeter of raw fiber;* D_1, *minor axis of mercerized fiber;* D_2, *major axis of mercerized fiber;* P_M, *perimeter of mercerized fiber.*

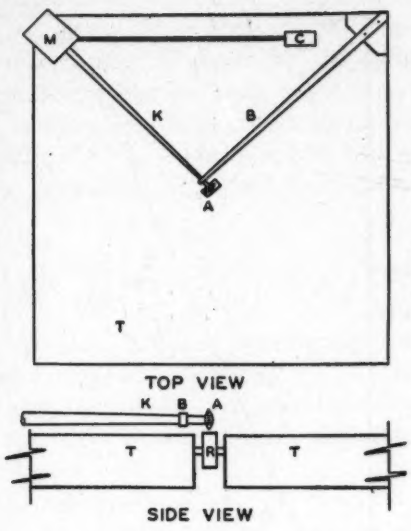

FIG. 2. *Diagram of instrument for measuring irregular linear distances.* A—*Knurled wheel.* B—*Brace.* C—*Counter.* K—*Drive shaft.* M—*Motor.* R—*Ball-bearing wheel.* T—*Table.*

the brace, B. A downward force of 1 to 2 lbs. is exerted by B on A to secure traction between it and the paper. Directly beneath A is a ball-bearing wheel, R (Figure 2, side-view diagram), which is countersunk in the table, T, and which projects above the surface of the table about 0.1 in. In operation, the desired line is followed by guiding the paper as it is drawn between A and R. The distance measured is read from a counter, C, which is also geared to the motor. The motor, M, is controlled by a foot-operated, start-stop switch. Owing to the tendency of the knurled wheel, A, to "dig-in," the instrument must be calibrated for a given grade of paper by following a regular line of known length.

Cross-sectional areas were measured with a polar planimeter by following the dashed lines (P_R and P_M in Figure 1). They include lumen area, estimated to be between 2% and 4% of the total area, introducing a small error which may be discounted. Experimental errors (sampling, preparation of the cross sections, and measurements) may amount to 5% of the area, as indicated by results on three duplicate bundles from the same sample. The error of measurement alone may amount to 2% of the area, as determined by three measurements of identical cross-section photomicrographs.

Assuming a statistical probability of 90% [1], the uncertainty in results was calculated to be in the neighborhood of 2% for perimeter and 4% for area.

Results

Results of the averaged 200 measurements on each sample are given in Table II, together with the calculated percent change in area, ΔA, percent change in perimeter, ΔP, and the two measures of shape: d_1/d_2 and $\frac{4\pi A_R}{P_R{}^2}$ [13]. The latter measure is preferred because of the relatively large error in measuring the small and poorly defined quantity d_1. Figure

TABLE II. CROSS-SECTIONAL DIMENSIONS OF FIBERS

Sample No.	Raw				Mercerized					
	Area, A_R (μ^2)	Perimeter, P_R (μ)	Shape d_1/d_2	Shape $4\pi A_R/P_R{}^2$	Area, A_M (μ^2)	Perimeter, P_M (μ)	Shape D_1/D_2	Shape $4\pi A_M/P_M{}^2$	ΔA (%)	ΔP (%)
1	152.9	52.1	0.40	0.71	174.8	48.5	0.71	0.93	14.4	7.4
2	141.8	51.0	.36	.68	153.8	45.9	.67	.92	8.4	11.3
3	123.8	48.2	.35	.67	144.0	44.2	.68	.93	16.3	9.0
4	94.2	42.0	.33	.67	110.9	38.6	.64	.94	17.8	9.0
5	120.5	47.9	.34	.66	136.0	43.1	.69	.92	12.9	11.1
6	115.3	48.0	.30	.63	125.3	43.1	.57	.85	8.6	11.5
7	122.9	49.8	.33	.62	138.5	44.0	.63	.90	12.8	13.4
8	126.6	50.6	.29	.62	135.7	43.2	.62	.91	7.2	17.1
9	170.4	58.9	.33	.62	184.5	50.8	.67	.90	8.3	15.9
10	155.0	56.9	.30	.60	174.9	50.0	.60	.88	12.9	13.8
11	131.0	52.6	.30	.60	159.2	47.3	.62	.89	21.6	11.1
12	118.6	50.5	.28	.58	129.6	44.0	.57	.84	9.3	14.9
13	160.3	59.0	.28	.58	173.1	49.6	.61	.89	8.0	19.1
14	151.0	57.3	.27	.58	167.7	49.8	.58	.85	11.0	15.0
15	135.0	54.3	.26	.58	149.6	46.9	.53	.86	10.8	15.8
16	126.4	55.2	.24	.52	147.1	46.6	.58	.85	16.3	18.5

3 shows that there is a good general relation between $\frac{4\pi A_R}{P_R^2}$ and d_1/d_2.

It is seen that upon mercerization without tension there is a wide variation in both percent change in area, ΔA, and percent change in perimeter, ΔP. Although each singly seemed to have no significant relation to the shape of the raw fiber, a relation is apparent between them (Figure 4) when the raw samples are divided into four groups according to shape $(4\pi A_R/P_R^2)$.

Partial correlation coefficients of percent changes in area, percent changes in perimeter, and $4\pi A_R/P_R^2$ also substantiate the interrelation of the three quantities:

ΔA *vs.* ΔP independent of $4\pi A_R/P_R^2 = -.78$
ΔA *vs.* $4\pi A_R/P_R^2$ independent of $\Delta P = -.67$
ΔP *vs.* $4\pi A_R/P_R^2$ independent of $\Delta A = -.96$

The low correlation of percent change in area with shape may be due to the unavoidably large errors inherent in mercerizing, sectioning, and measuring the fiber sections. Thus, the variation in this value may be actually considerably less than is indicated.

Discussion

Geometric Relations

The relation of percent changes in area and in perimeter to the shape of the raw fiber may be understood better if it is partly explained as being due to a change toward circularity. Such changes result from an increase in area or a decrease in perimeter or both. The percent increase in area is defined as

$$\Delta A = 100\left(\frac{A_M - A_R}{A_R}\right). \qquad (1)$$

In order to obtain an equation which is a simple function of shape, it is necessary to define the percent change in perimeter as

$$\Delta P = 100\left(\frac{P_R - P_M}{P_M}\right). \qquad (2)$$

This equation is related to the usual concept in which the perimeter of the raw fiber is used as a base, by the factor P_R/P_M. This alters the magnitude of the values but does not alter their relative order.

On this basis, a general equation relating the change in shape to the changes in area and perimeter which can be applied to any shape area is derived:

$$\left(1 + \frac{\Delta A}{100}\right)\left(1 + \frac{\Delta P}{100}\right)^2 = \left(\frac{4\pi A_M}{P_M^2}\right)\left(\frac{P_R^2}{4\pi A_R}\right). \qquad (3)$$

If the final (mercerized) shape is circular, the term $\frac{4\pi A_M}{P_M^2}$ becomes 1, and if there is also no change in perimeter this equation would illustrate one case discussed by Skau [14].

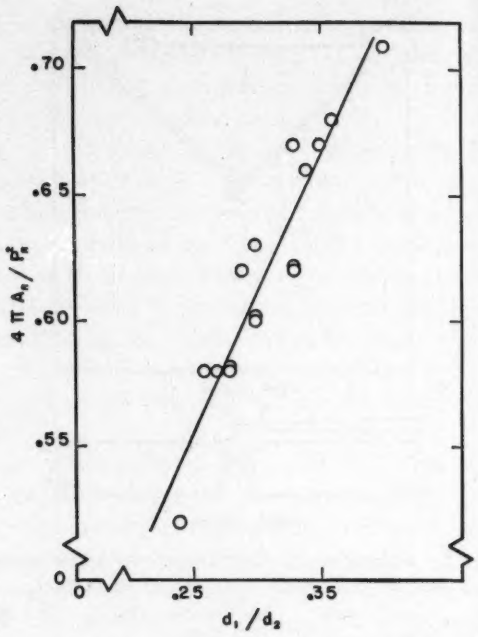

FIG. 3. *Relationship between two shape factors of the raw fiber.*

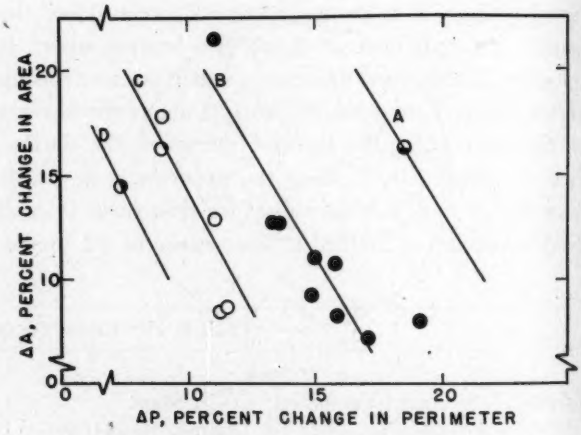

FIG. 4. *Relationship between area change and perimeter change upon mercerization without tension. Curves* A, B, C, *and* D *are those for shape factors* $(4\pi A_R/P_R^2)$ *of .52, .60, .65, and .71, respectively.*

The shape of the mercerized fibers was never circular (see Table II). Their shape was, however, relatively constant, as shown by the coefficients of variation determined on mean shape values for the sixteen samples—for mercerized fibers, 3.70%; for raw fibers, 7.85%. Therefore, if the mercerized shape is considered as constant, equation (3) reduces to

$$\left(1 + \frac{\Delta A}{100}\right)\left(1 + \frac{\Delta P}{100}\right)^2 = K \Big/ \frac{4\pi A_R}{P_R^2}. \quad (4)$$

The curves A, B, C, and D in Figure 4 were drawn using an average value of $4\pi A_R/P_R^2$ in equation (4). The observation of the greatest dimensional changes in the flat fibers is in accordance with equation (4).

Practical Interpretation

Since fibers of different shapes differ in their characteristics upon mercerization without tension, differences would be expected if routine mercerization practices were applied to cottons of all shapes. Swelling with caustic solution without tension is complicated by the tensional forces developed in yarns or fibers held at a definite length during the commercial process. Tension applied to fibers during mercerization allows a smaller increase in area and increases the circularity over that of fibers mercerized without tension [8]. Thus, an even greater decrease in perimeter occurs upon mercerization with tension. As there is little change in fiber length in mercerization with tension, there is a considerable decrease in surface area of the fiber due to the decrease in perimeter. This decrease in surface area, together with the increased circularity, should increase the luster and reduce the soiling properties of the flat (immature) more than of the round (mature) fibers.

Conclusions

The variation in shape of the mercerized samples was less than that of the raw samples. Samples composed of flat (immature) fibers generally shrank more in perimeter than did thick-walled (mature) samples.

Two methods of measuring shape, d_1/d_2 and $\frac{4\pi A_R}{P_R^2}$, agreed.

Acknowledgment

Appreciation is expressed to Mary L. Rollins for valuable suggestions and discussions; to Ines V. deGruy for preparation of the cross sections and photomicrographs; to C. A. Caillet, D. H. Walsh, and Mary W. Rhodes for the cross-sectional measurements; and to Dorothy C. Legendre and Annie B. Cooper for the measurements of physical properties.

Literature Cited

1. American Society for Testing Materials, Manual on Presentation of Data, Supplement A, 1935.
2. American Society for Testing Materials, Committee D–13, Tentative General Methods of Testing Cotton Fibers (D 414–47 T), 1938, p. 219.
3. American Society for Testing Materials, Committee D–13, Tentative General Methods of Testing Cotton Fibers (D 414–47 T), 1938, p. 228.
4. Anderson, D. B., and Kerr, T., *Ind. Eng. Chem.* **30**, 48 (1938).
5. Balls, W. L., *Proc. Roy. Soc.* (*London*) **B95**, 72 (1923).
6. Calvert, M. A., and Summers, F., *J. Textile Inst.* **16**, T233 (1925).
7. Coward, H. F., and Spencer, L., *J. Textile Inst.* **4**, T32 (1923).
8. Giuffria, R., and Tripp, V. W., TEXTILE RESEARCH JOURNAL **22**, 21 (1952).
9. Goldthwait, C. F., Smith, H. O., and Barnett, M. P., *Textile World* **97**, 105 (1947).
10. Karrer, E., and Bailey, T. L. W., Jr., TEXTILE RESEARCH **8**, 381 (1938).
11. Marsh, J. T., and Wood, F. C., "Introduction to Chemistry of Cellulose," London, Chapman Hall, 1938, p. 63.
12. Moore, A. T., Scott, L. W., deGruy, I. V., and Rollins, M. L., TEXTILE RESEARCH JOURNAL **20**, 620 (1950).
13. Schloemer, H., *Kunstseide* **12**, 380 (1930).
14. Skau, E. L., TEXTILE RESEARCH JOURNAL **16**, 556 (1946).
15. Willows, R. S., and Alexander, A. C., *J. Textile Inst.* **13**, T237 (1922).

(*Manuscript received October 29, 1951.*)

Letters to the Editor

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A Method of Determining the Concentration Dependence of the Diffusion Coefficient

UNIVERSITY OF KYOTO
Department of Fisheries
Maizuru, Japan
November 27, 1951

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

With reference to the diffusion of material in high polymeric substances, attention has recently been concentrated on the calculation of the dependence of the diffusion coefficient upon the concentration of diffusing material from experimental sorption data. Thus, Crank *et al.* [2] have developed a method of successive approximations, and Prager [4] has used a method of approximating the actual concentration dependence by a step function. These methods are rather involved in practical applications; a different and somewhat simpler procedure is presented herein.

As shown in Prager's paper, for a film of area A normal to the direction of diffusion, the sorbed mass in the film, Q, may be expressed by the equation

$$Q = 2A\sqrt{t}K(C_f), \tag{1}$$

if the time, t, is short and if the resistance to material transfer at the film surface (where the film is in contact with the solution of concentration C_f) may be neglected. Here $K(C_f)$ is a function of C_f to be determined if the form of $D(C)$ is known.

Some time ago, one of us [3] proposed an approximate method of solving one-dimensional diffusion equations involving the diffusion coefficient with an arbitrary concentration dependence based on Yamada's idea [5] for dealing approximately with the laminar boundary-layer equation. According to this method, the form of $K(C_f)$ may be expressed approximately by

$$K(C_f) = \sqrt{D(O)}C_f\left(\frac{1}{8\sqrt{\beta}} + \frac{1}{12}\alpha\sqrt{\beta}\right), \tag{2}$$

where

$$\alpha = \frac{30}{C_f}\int_0^{C_f}\frac{D(C)}{D(O)}\,dC, \tag{3}$$

and β is a larger positive root of the quadratic equation

$$24\alpha\frac{D(C_f)}{D(O)}\beta^2 + \left\{\alpha - 108\frac{D(C_f)}{D(O)}\right\}\beta + \frac{3}{2} = 0. \tag{4}$$

The accuracy of equation (2) is chiefly dependent upon the form of $D(C)$. A detailed discussion of this point will be made in a future work.

The actual form of $K(C_f)$ may be obtained experimentally; our problem is to calculate $D(C)$ from this experimental information by making use of the equations given above.

From equation (2) we have

$$\sqrt{D(O)} = \lim_{C_f\to 0}\left\{\frac{K(C_f)}{C_f}\left(\frac{1}{8\sqrt{\beta}} + \frac{1}{12}\alpha\sqrt{\beta}\right)^{-1}\right\}. \tag{5}$$

From equations (3) and (4) we see that $\alpha \to 30$ and $\beta \to \frac{1}{12}$ as $C_f \to 0$. Thus, the value of $D(O)$ may be obtained from the relation

$$D(O) = \left\{\frac{1}{1.155}\lim_{C_f\to 0}\frac{K(C_f)}{C_f}\right\}^2. \tag{6}$$

Next, we assume a polynomial representation for $D(C)$:

$$D(C) = D(O)(1 + k_1C + k_2C^2 + \cdots). \tag{7}$$

The unknown coefficients, k_1, k_2, etc., may be determined by fitting the plot of $K(C_f)$ as obtained from equations (2), (3), and (4) (after substituting equation (7) into them) at suitably selected points of C_f. The number of points must be equal to the number of coefficients to be determined.

In some cases, it is also of practical interest to approximate the actual form of $D(C)$ by an exponential representation such as:

$$D(C) = D(O) \cdot \exp(\lambda C), \qquad (8)$$

with one parameter, λ. This form of concentration dependence of the diffusion coefficient has recently been illustrated by Boyer [1] on a polystylene-chloroform system; further evidence is found in Prager's results on an isobutane-polyisobutylene system.

In order to check our method, we shall apply it to Prager's system by assuming the exponential dependence on diffusion coefficient indicated in equation (8).

First, the value of $D(O)$ is found to be 1.50×10^{-9} cm.²/sec. Next, we determine the value of λ in such a way that $K(C_f)$ given by equation (2) coincides exactly with the corresponding experimental value at $C_f = 0.0182$ g./cc. We then have $\lambda = 44.9$ cc./g. Substituting these values for $D(O)$ and λ into equation (8) gives the results listed in the second column of Table I. In the third column are given the corresponding results computed by Prager. Agreement between these two sets of results is fairly good.

Finally, using the values of $D(O)$ and λ obtained above, we recalculate the value of $K(C_f)$ from equation (2) and subsequent equations, and compare the results with the corresponding experimental values obtained by Prager. This comparison is made in the fourth and fifth columns of Table I. It is seen that our calculations satisfactorily reproduce the observed values for the isobutane-polyisobutylene system.

TABLE I

C_f (g./cc.)	$D\times10^9$ (cm.²/sec.) (Authors)	$D\times10^9$ (cm.²/sec.) (Prager)	$K\times10^6$ (g./cm.² sec.$^{\frac{1}{2}}$) (Calculated)	$K\times10^6$ (g./cm.² sec.$^{\frac{1}{2}}$) (Observed)
0.0000	1.50	1.8	0.00	0.00
0.0091	2.25	2.3	0.46	0.45
0.0182	3.40	3.4	1.05	1.05
0.0273	5.10	5.1	1.81	1.79
0.0364	7.65	7.4	2.74	2.73
0.0455	11.5	10.3	3.99	3.93

A similar analysis based on the polynomial approximation of $D(C)$ will be reported elsewhere.

Literature Cited

1. Boyer, R. F., *J. Polymer Sci.* **5**, 139 (1950).
2. Crank, J., *et al.*, *Trans. Faraday Soc.* **45**, 240, 636, 1119 (1949).
3. Fujita, H., *Mem. Coll. Agr., Kyoto Univ.* **59**, 31 (1951); *Bull. Japan. Soc. Sci. Fish.* (in press).
4. Prager, S., *J. Chem. Phys.* **19**, 537 (1951).
5. Yamada, H., *Rept. Research Inst. Fluid Eng.* **3**(3), 29 (1947).

HIROSHI FUJITA
AKIRA KISHIMOTO

Fatigue of Tire Cords as a Function of Twist

STÉ AME FABELTA
Usine de Zwijnaarde
Ghent, Belgium
September 13, 1951

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

A good tire cord must fulfill three essential conditions: it must exhibit high tenacity, low elongation, and good resistance to fatigue.

A single yarn having these essential features does not necessarily give a good cord because twist and tension during twisting contribute greatly to the final characteristics of the cord.

A great deal of research has been devoted to the study of the influence of ply and cable twist on the static and dynamic properties of cords. In the article "A Fatigue Test for Tire Cords," Chester B. Budd * concludes that "overtwisting lowers the cord tensile, but improves the flex life."

* TEXTILE RESEARCH JOURNAL 21, 174–9 (Mar. 1951).

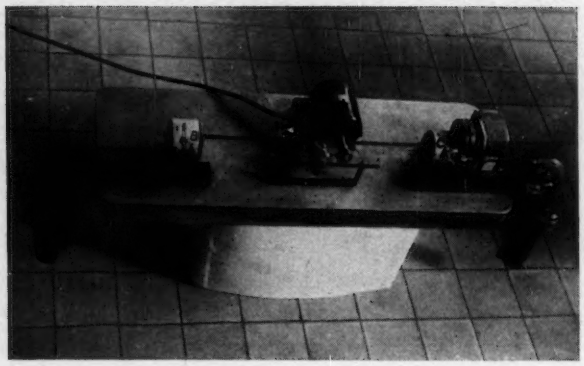

FIG. 1. *Fatigue testing apparatus.*

However, in measuring flex life, Budd uses the same static load independent of the twist, and thus independent of the cord size. But an increase in twist causes contraction, and this increases the size of the count; and applying the same static load in fatigue tests acts to the advantage of higher twist. In order to decide whether the load should be chosen identical to or proportional to the cord count, the manufacture of a tire should be considered. When making a tire-cord fabric, the problem is to satisfy the requirements with the least weight and volume of cord. Thus, there is no doubt that the cord size has to be taken into account. It is nearer to reality to choose a static load that is proportional to the cord size rather than the same static load for different sizes.

Applying those principles, we have measured the fatigue life of a series of cords with ply and cable twist varying from 1 to 6 turns per cm. (2.5 to 15 turns/in.) with the following combinations: (1) the same ply and cable twist; (2) ply twist with 1 turn/cm. more than cable twist; (3) ply twist with 1 turn/cm. less than cable twist.

The fatigue life was measured with the apparatus shown in Figure 1. This apparatus works with the following constants: stroke, 1.8%; frequency, 25 per sec.; distance between clamps = AB = AC = 25 cm.; static load on clamps B and C, the weight of 13 km. of the measured cord.

The fatigue life is expressed by the mean number of kilocycles endured by the cord at the moment of break.

The total number of measurements, n, is fixed by the condition that

$$\frac{t_{0.05}}{\sqrt{n-1}}\, V\% < 25\%,$$

where $t_{0.05}$ is the value of Students "t" at a probability level of 0.05 and $V\%$ is the coefficient of variation.

The measurements were carried out in standard atmosphere (65% ± 2% R.H., 20° ± 2°C). The cord size in tex was determined with 13% regain.

Figure 2 plots the cord size in tex (g./km.) and the fatigue life in kilocycles against ply twist for different

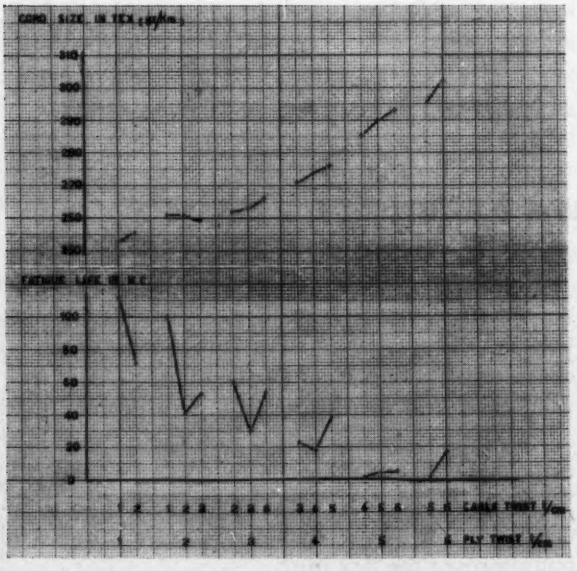

FIG. 2. *Effects of increasing ply twist on cord size and fatigue life.*

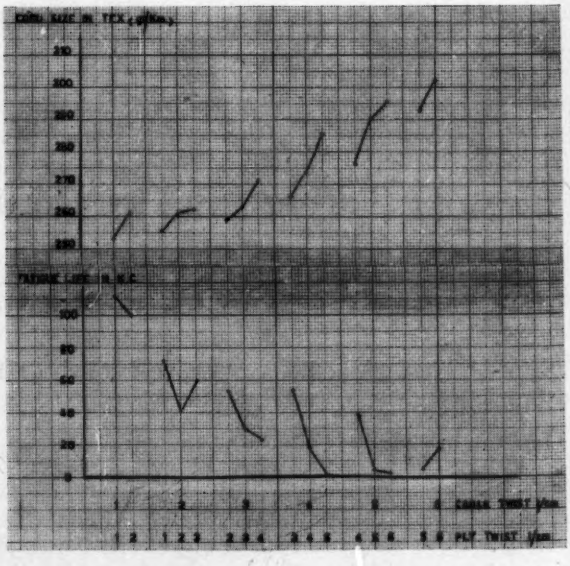

FIG. 3. *Effects of increasing cable twist on cord size and fatigue life.*

cable twists. Figure 3 is a plot of the same characteristics against cable twist for different ply twists.

Both figures prove that, on the basis of static load being proportional to cord size, overtwisting lowers the fatigue life.

L. HERMANNE
G. QUINTELIER

Author's Comments

In the paper "A Fatigue Test for Tire Cords," Figure 4 presents the effect of increasing cable twist on fatigue life as measured on the fatigue tester which is the subject of the above paper. The graph indicates that "overtwisting lowers the cord tensile, but improves the flex life." Messrs. Quintelier and Hermanne take issue with this statement because no adjustment in static load was made for increases in cord size. Their contention is that if an adjustment had been made, the conclusion would be reversed. They then submit data, obtained with a fatigue tester of their own design, in support of their contention.

The cords used in Figure 4 ranged in size from 2.34 to 2.18 (based on the cotton system) for the 6 t.p.i. in the ply, and from 2.37 to 2.23 for the 9 t.p.i. in the ply. The corresponding tensiles were 15.9 to 11.9 lbs. and 14.8 to 13.4 lbs., respectively. The static load was 3.1 lbs. If an adjustment for cord size had been made, the larger cords should have been tested under approximately 3.3 lbs. A glance at Figure 8 ("effect of static load on fatigue life of tire cord") indicates that such a change in load would have had too small an effect on the results obtained to necessitate any modifications of the conclusion that overtwisting improves flex life.

It is dangerous to make direct comparisons between two different fatigue testers in the hope that both will yield the same results. Even if the two tests, that of Quintelier and Hermanne and that of the present author, are presumed equivalent, the data of Figure 2 (Quintelier and Hermanne) does not negate, but rather strengthens, the statement made about overtwisting. If the ply twist is fixed and the cable twist is progressively increased beyond the ply twist, their data (Figures 2 and 3) show an increase in fatigue life. The term "overtwisting" was not intended to mean a twist structure of greater magnitudes in the ply and cable construction, but rather an increase of cable twist considerably beyond that in the ply; this was implied in Figure 4 of the original report.

C. B. BUDD
The B. F. Goodrich Company
Brecksville, Ohio

(November 8, 1951.)

The Visibility of the "Cuticula" of Wool Under the Phase-Contrast Microscope

FIBRE RESEARCH INSTITUTE
Delft, Holland
November 14, 1951

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

In 1948, Lindberg, Philip, and Gralén [1] discovered by means of electron microscopy the existence of a thin membrane of about 100 Å. thickness covering the surface of animal hair.

Schuringa and Algera [2], who also used the electron microscope, confirmed this observation and showed that the membrane is identical with the wall of the Allwörden sac. It was suggested that the name "epicuticula" be replaced by "cuticula."

Continued research proved that the membrane is also observable under the phase-contrast microscope. Degreased wool was treated for 20 min. in a saturated solution of bromine. After rinsing out the bromine, the wool was agitated by hand in a small amount of distilled water, and thus a suspension of fragments of the Allwörden membrane was obtained. A drop of this suspension was deposited on the collodium

FIG. 1. *Wool "cuticula." Eyepiece lens 8 ×, phase-contrast object lens 12 ×. (Photographically magnified to 1000 ×.)*

film of a specimen holder of the Philips electron microscope belonging to the Technical Physical Service T.N.O.

After evaporation of the water, photomicrographs of the preparation were made with a Leitz Panphot in which the original Leitz microscope was replaced by a Wild phase-contrast microscope (without the binocular tube assembly).

Figure 1 shows four of the fragments of membrane photographed. Three of them are more or less rolled up, but the fourth is expanded.

In order to demonstrate that the objects shown in Figure 1 were actually membranes, the preparation was shadowcast with gold-manganin at an inclination of 1 : 5, and an electron photomicrograph was taken of the membranes shown in Figure 1 (Figure 2).

These membranes exhibit the properties of the

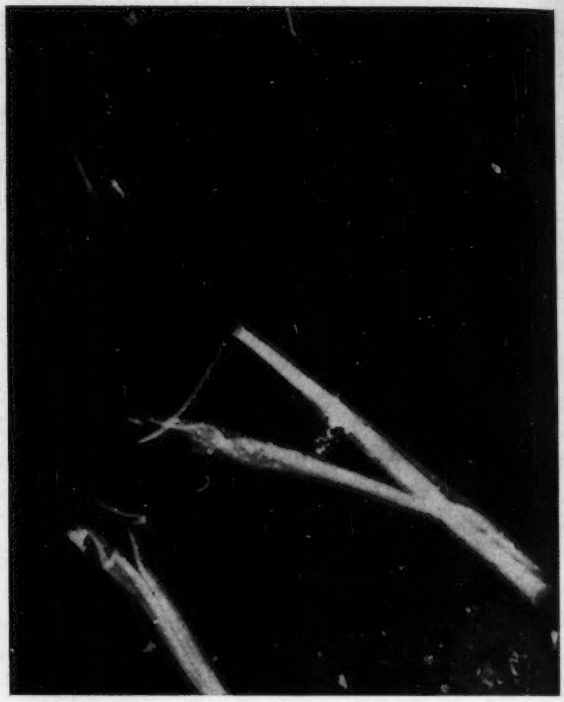

FIG. 2. *Electron photomicrograph of the same "cuticula" fragments as shown in Figure 1; shadowcast with gold-manganin at an inclination of 1 : 5. (Magnification, 1500 ×.)*

"cuticula," which appears, among others, from their thickness being 100 Å.*

* While this letter was being prepared, an article by P. F. Mariner appeared in *Nature* **167**, 231–2 (1951) which also treats the visibility of the "cuticula" under the phase-contrast microscope.

Literature Cited

1. Lindberg, J., Philip, B., and Gralén, N., *Nature* **162**, 458–9 (1948).
2. Schuringa, G. J., and Algera, L., *Biochim. et Biophys. Acta* **6**, 325–33 (1950).

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INDUSTRIAL SECTION

A High-Speed Recording Yarn Tensiometer*

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Abstract

A yarn tensiometer is described which is capable of detecting and recording variations in tension occurring at a rate of the order of 100 cycles per sec. Tension variations of a considerable magnitude at such frequencies have been shown to exist in yarn running on standard textile machinery.

THE MEASUREMENT and proper regulation of yarn tension is of recognized importance in the textile industry. It is understandable, therefore, that a great number of devices have been used in an attempt to study yarn tension variations. The most common tensiometers are mechanical, employing some type of spring member, the deflection of the spring being indicated by a pointer on a dial. Many of these mechanical tensiometers are built with a high degree of precision and are calibrated accurately. However, the large movement necessary to give an accurate tension indication and the inertia of the system necessarily make the response of such tensiometers slow. Since extremely rapid variations in running yarn tension do occur in many textile operations, and it is well known that rapid variations in tension cannot be seen on these tensiometers, they cannot be used for research in this field. For much research on the operation of textile machinery, a record is required of tension variations as they actually occur in running yarn. Study has shown that in some cases these variations are of a large magnitude as compared with the average tension measured by tensiometers now available. In order to record all yarn tension variations as they are actually occurring, a tensiometer and recorder with a high speed of response must be employed.

Mechanical devices have obvious limitations and, therefore, in order to accomplish this the most easily, it seems evident that the tension variations should be converted into electrical signals and these then recorded on existing recorders. Several schemes have been employed successfully for doing this [1, 4]. However, the mechanical linkages used in these tension transducers seriously lower the frequency response.

A Tensiometer with Medium Speed of Response

The study of recording tensiometers was begun at this laboratory a number of years ago. At that time the use of strain gages for instrumental work was a relatively new practise, but the small movement necessary to obtain considerable electrical output from these devices indicated that a high speed of response could be obtained with proper design. Research was carried out with ordinary bonded resistance strain gages mounted on a steel beam with a pulley at the end of the beam. However, when the General Electric Magnetic Strain Gage and power unit became available, the bonded type was aban-

* Adaptation of talk given by Mr. Nistico before a meeting of The Fiber Society at Atlanta, Ga., Apr. 19, 1951.

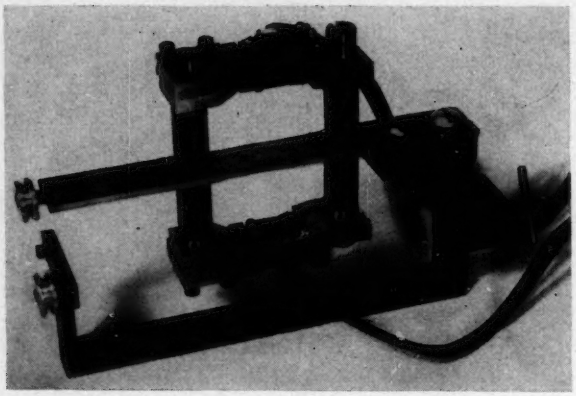

FIG. 1. *Magnetic strain gage tensiometer.*

doned because the magnetic type offered a simple way of obtaining a sensitive instrument with a fairly rapid frequency response.

The General Electric Magnetic Strain Gage is of the variable-reluctance type. The gage consists of a coil mounted on an iron core, with an air gap in the iron core. Variations of the width of the air gap cause variations in the reluctance path and, hence, variations in the impedance of the coil. These variations in impedance are then translated into an electrical signal. Figure 1 shows two such gages mounted back to back on a steel beam to form a tensiometer. One pulley is fixed to the frame and another is mounted on the steel beam carrying the gages. This steel beam is of such a design that its natural vibrational frequency is high enough so as not to be recorded on the recorder chosen. The gages are mounted at a distance from the beam in order to obtain enough output for sensitive calibrations. Such a mounting does not seriously affect the linearity of the system.

The two coils of the gages are essentially in a bridge balanced by two 5 mfd. capacitors. Sixty-cycle current is fed to the gages through a power unit supplied with the gages by the manufacturer. The output of the bridge is rectified and filtered in the power unit, and can be fed directly to a recording microammeter without amplification. The frequency response of this equipment is of the order of 6 cycles per sec.

Figure 2 shows the complete equipment set up to record tension variations. Figure 3 illustrates a practical application of the device. It shows a tension chart made by running yarn off a headless bobbin at a constant linear speed, the tension being due only to the runoff from the bobbin and a fixed guide. This bobbin of yarn was produced on machinery where different portions of the traverse action made complete cycles in 100 sec. and 11 sec., respectively. Each curved division on chart *1* represents 50 sec. of time. The centers of the groups of tension peaks occur every two divisions, or 100 sec. apart. Chart *2* is the same as chart *1* except that the recorder chart speed is increased so that each curved division represents $\frac{5}{6}$ sec. The distance between the peaks on this chart represents about $5\frac{1}{2}$ sec., which corresponds to half of the 11-sec. cycle previously mentioned.

It may be noted from chart *2* that minute variations appear to occur within the $\frac{5}{6}$-sec. intervals, indicating

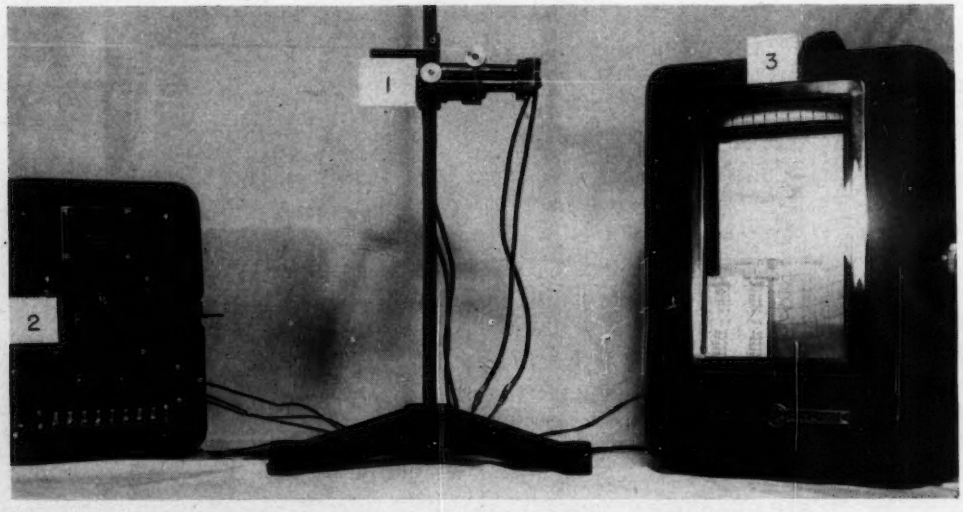

FIG. 2. *Magnetic strain gage tensiometer and recorder.* 1—*Tensiometer.* 2—*Power unit.* 3—*Recorder.*

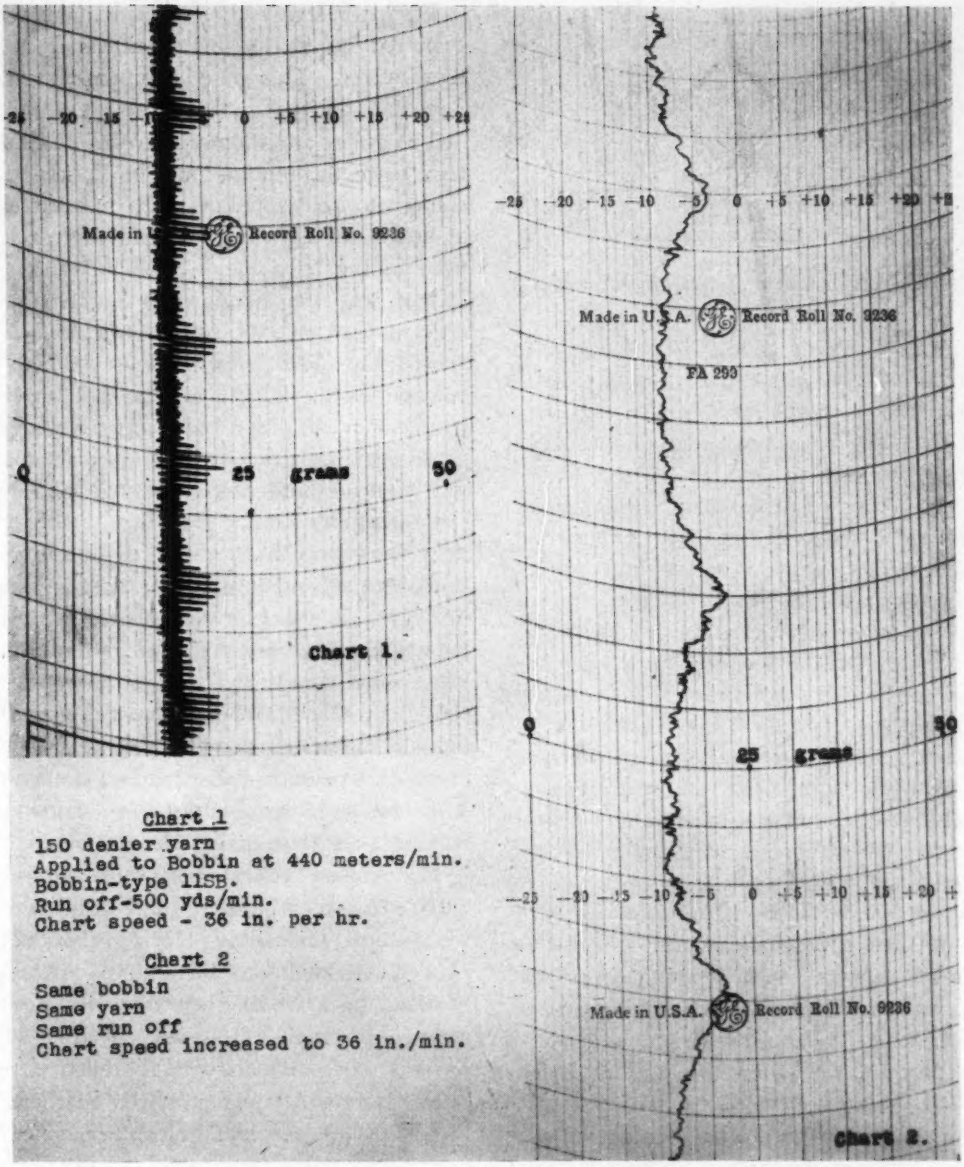

FIG. 3. *Tension variations measured with magnetic strain gage tensiometer.*

that since the frequency response of this particular equipment is of the order of 6 cycles per sec., much larger variations in tension could be occurring in this interval than would be recorded on the chart. For example, when this bobbin was wound, the traveller was going at the rate of about 30 revolutions per sec. If there had been improper lubrication of the ring at one spot or a mechanical imperfection which produced excessive friction, then variations in tension at a rate of 30 cycles per sec. might be occurring. These would show up as a small fraction of the actual magnitude, or might not be visible at all if the over-all frequency response of the equipment were such that it dropped off to zero before this frequency was reached.

A Tensiometer with High Speed of Response

It is important that tension variations occurring at high frequencies be recorded and studied. For this purpose an instrument was developed with a higher frequency response. As a first approximation, it was assumed that frequencies of the order of 30 to

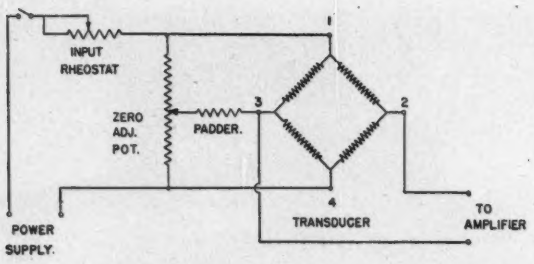

FIG. 4. *Electrical connections of Statham transducer.*

60 cycles per sec. would be as high as one would encounter in most textile operations. However, after some experience in this field it was believed that a flat response to 100 cycles per sec. and possibly higher would be desirable.

Magnetic strain gages and auxiliary equipment are available for high-frequency response. However, when these are mounted on a beam as shown for the preceding tensiometer (Figure 1), the natural period of vibration of the beam is a limiting factor, and the balance between sufficient movement of the gages for observable output and stiffness of the beam for high natural period of vibration is difficult.

It was therefore decided to develop some other device for converting high-speed tension variations into electrical impulses. After considerable research, a Statham model G-4-225 force transducer having a 4-oz. full-load range was finally chosen. This device is manufactured by the Statham Laboratories, Beverly Hills, Calif., and consists of four unbonded resistance elements mounted on a flexible beam, all being equally prestressed so that they are under tension. As the beam is flexed, the tension increases on two of the elements and decreases on the other two. The arrangement of the four elements of the transducer allows all four to act as strain gages, so that they can be wired directly in a bridge circuit, with four times the output of a single element.

Figure 4 shows the electrical connection of the four elements. These are connected internally in the transducer, and only four terminals are brought out, these being marked *1* to *4* in Figure 4. In actual use, it is only necessary to connect the input to terminals *1* and *4*, and take the output from *2* and *3*. The padder and zero-adjustment potentiometer may be used to balance out any initial loading on the transducer so that the calibration may be started at zero on the recorder. The power supply for this transducer must be able to supply a maximum of 7 v. and 30 milliamps., either A. C. or D. C.

Figure 5 shows the Statham gage with a pulley mounted on its loading stem, as it is used for a tensiometer. This gage is compact, measuring about 2 in. × 1½ in. × ¾ in.

The pulley and mount offered a small design problem, since the mount must be light and yet sturdy enough to hold a pulley with yarn travelling over it at speeds of 500 m./min. and higher. The pulley used in the tensiometer with medium speed of response was the standard ball-bearing type that is used in most mechanical tensiometers. At high yarn speeds, the added tension due to the high friction developed in these pulleys is considerable. For the tensiometer with high speed of response, pulleys were made with integral shafts ending in conical points. The conical shafts were mounted in Miniature Precision Angular Contact Bearings, made by the Miniature Precision Ball Bearing Co., Keene, N. H. With these bearings mounted in the closed yokes, as shown in Figure 5, added tension due to pulley friction was kept below 0.1 g. per pulley at yarn speeds of 500 m./min. This may be determined by means of two tensiometers which previously have been calibrated individually. The two are then run in series, and the increase in tension in the second tensiometer measured. This tension increase is due to the friction in the pulleys between the sensing elements.

The closed yokes make threading-up difficult. Open yokes may be used, the bottom two pulleys being mounted on a sliding block so that the tensiometer may be inserted into a running yarn system. The Statham gage would then work in compression instead of tension. However, accurate pulley alignment is more difficult with the open yokes.

The natural frequency of the Statham transducer is 450 cycles per sec. However, since the pulley mount and pulley are directly connected to the weighing system, this added weight lowers the natural frequency of the gage. The gage element itself weighs 4.3 g., and the pulley and mount unit weigh 10.8 g. Since the natural period of vibration of a spring is in-

FIG. 5. *High-speed tensiometer.*

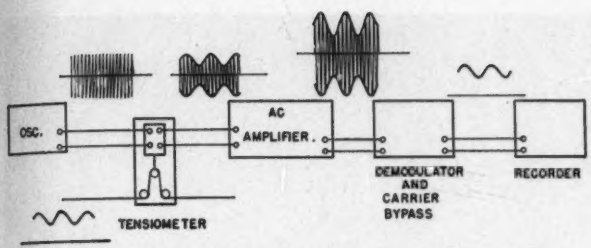

FIG. 6. *Block diagram of carrier system for amplifying and recording tension variations.*

versely proportional to the square root of the mass hung on the spring, the natural frequency of the gage with the pulley and mount connected is

$$f_2 = 450\sqrt{\frac{4.3}{4.3 + 10.8}} = 260 \text{ cycles per sec.}$$

This is considerably higher than the frequencies to be detected by the tensiometer, and, accordingly, the gage is usable for the desired purpose.

A Brush Direct Inking Oscillograph was chosen as a recorder. In order to drive this recorder, considerable amplification is necessary. It is also necessary that the amplification be flat from D. C. to the upper frequency limit of the recorder. This may be accomplished with a high-grade D. C. amplifier, but the inherent instability of this type of amplifier together with other well-known difficulties involved in their use led to the use of the carrier-frequency principle. Figure 6 is a block diagram of such a system. An oscillator delivering alternating current at a constant frequency between 1000 and 2000 cycles per sec. is used as the power source for the strain gage transducer, this being the carrier frequency. Upon this carrier frequency are impressed electrical impulses from the output of the transducer, these later being equivalent to the tension variations occurring in the travelling yarn. The modulated carrier wave thus obtained is amplified in an A. C. amplifier. The amplified signal is then demodulated to remove the carrier frequency, and the remaining electrical signal is then recorded, producing a trace of yarn tension.

In the design of such a system it is desirable to make the frequency response of the amplifier such that it will correct the response of the recorder and therefore increase the frequency response of the whole system. The Brush Development Company produces the Universal Analyzer Model Bl-320 with a self-contained oscillator which is designed specifically for use with strain gages to work into the Brush recorder. Although some of the earlier work was done with amplifiers designed in this laboratory, this Universal Analyzer is now used exclusively.

The Brush Direct Inking Oscillograph has a frequency response which begins to fall off at 20 cycles per sec., and is down 3 db at 60 cycles per sec. The Universal Analyzer has a response which begins to rise at 20 cycles per sec., continues to rise up to 120 cycles per sec., and then falls off rapidly. When this latter instrument is used with the Brush oscillograph, the frequency response of the system is very nearly flat to 100 cycles per sec., and falls off 3 db at 120 cycles per sec. With this frequency response it is possible to record variations in tension in running yarn caused by machine parts which are rotating as fast as 6000 r.p.m. and to obtain a part of the variation for rotations as fast as 7200 r.p.m.

To calibrate the tensiometer, yarn threaded through the pulleys was fixed at one end and a dead weight loaded at the other end. A suitable additional pulley was used so that calibration could be effected with the tensiometer in the vertical position.

Experimental Data

To demonstrate the use of the tensiometer in a textile operation, a ring spinning apparatus was chosen. The bobbin used on this equipment was $1\frac{3}{4}$ in. in diameter, 8 in. in length, and had $3\frac{1}{4}$-in. flanges. The ring rail was 4 in. in diameter. Cellulose acetate yarn of 150 denier, 40 filament, and $\frac{1}{2}$ turn per in. was used in all of the following experiments.

The tensiometer was interposed between the feed roll and balloon guide of the ring spinning takeup unit so that the tension variations in the balloon could be measured during the winding of a bobbin. Figure 7 shows two views of the ring spinning frame with the tensiometer in place. This spinning frame was specially constructed for experimental purposes, with independent, adjustable drives for the spindle and the feed roll. The balloon guide above the spindle was normally stationary while the traveller ring traversed the spindle. However, for experimental purposes, the balloon guide could be fixed to the traveller ring so that it moved with the ring and kept the balloon length constant.

With yarn running at a feed rate of 50 m./min., a spindle speed of 5240 r.p.m., and the balloon guide stationary, the tension records shown in Figure 8 were produced. The top chart was obtained with

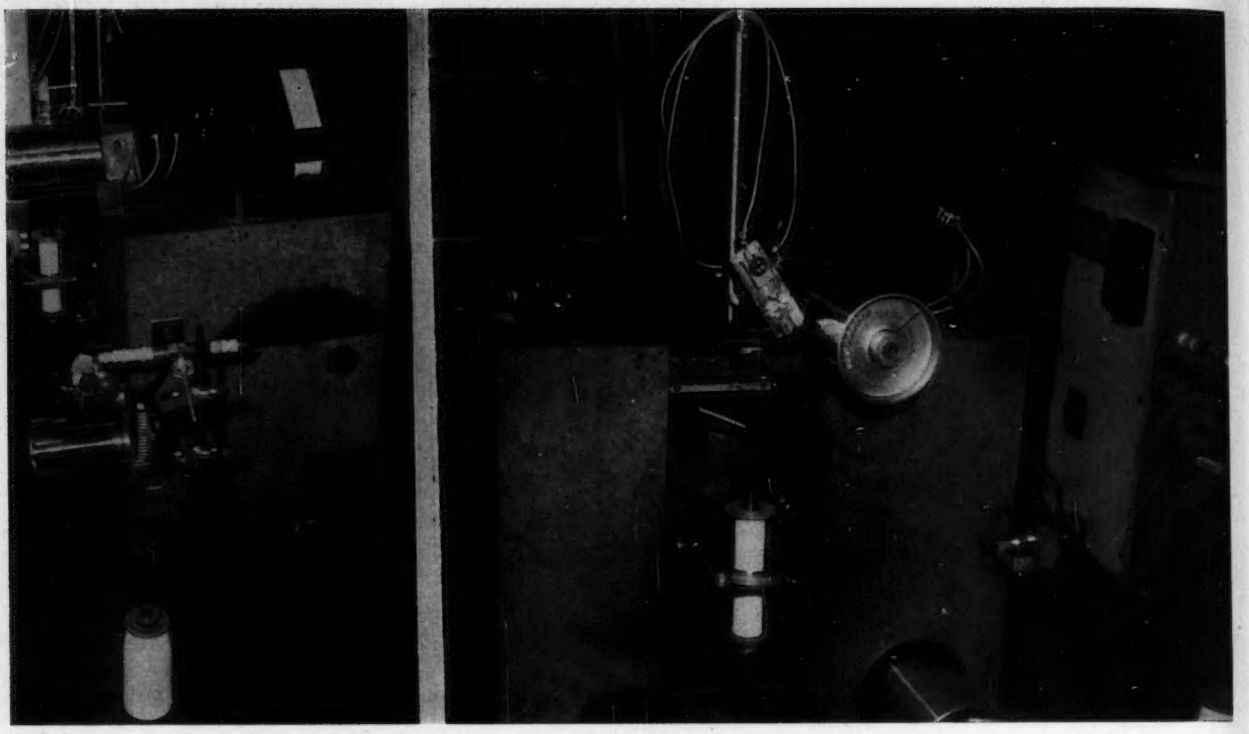

FIG. 7. *Two views of ring spinning frame with tensiometer in place.*

the General Electric magnetic tensiometer, whereas the middle and bottom charts were obtained with the high-speed tensiometer. It is evident that the magnetic tensiometer does not show the high-frequency tension peaks which are picked up by the high-speed tensiometer. Of particular interest is the portion where the tension variation becomes very high. This occurred when the ring rail reached the top of the traverse stroke, as marked by the arrows in the charts of Figure 8. From the bottom chart it may be seen that before the high tension peaks occur, a uniformly repeating pattern of tension variation is evident. The frequency of this tension variation is 81.5 cycles per sec. As the high peaks of tension appear, the pattern changes somewhat, the tension rising to a high peak during one cycle and then barely rising at all on the next cycle. Although the average tension during the entire traverse remains fairly constant at about 12.5 g., the high-speed tensiometer shows that the tension actually varies from a low of about 8 g. to a high of about 15 g. during each cycle until the top of the traverse stroke approaches. The tension then varies from 5 to 25 g. during one cycle for a period of about 1 sec. Possible reasons for these variations in tension will be mentioned later.

The large high-frequency tension variation occurring at the top of the traverse stroke appeared to be of sufficient interest to warrant further investigation. Figure 9 shows records of the balloon tension at various yarn feed rates, with a spindle speed of 5240 r.p.m. The frequency of the tension variations is seen to vary with the yarn feed rate.

The change in tension frequency with changing feed rate suggested that the tension variations might be associated with the traveller speed. The traveller speed was calculated * for each set of running conditions, and the results are summarized in Table I.

The good agreement between traveller speed and frequency of yarn tension variation is convincing evidence that there is a variation in friction or some other force between the bobbin and the balloon guide which goes from a maximum to a minimum for each revolution of the traveller on the ring.

In an effort to learn more about tension variation as a function of the traverse stroke, the yarn take-up

* Traveller speed may be calculated as follows [3]:

$$f = b - \frac{v\,(39.37)}{d}\,(60)$$

where: v = yarn feed rate, m./min.; f = traveller speed, r.p.s.; b = bobbin speed, r.p.s.; d = bobbin winding on diameter, in.

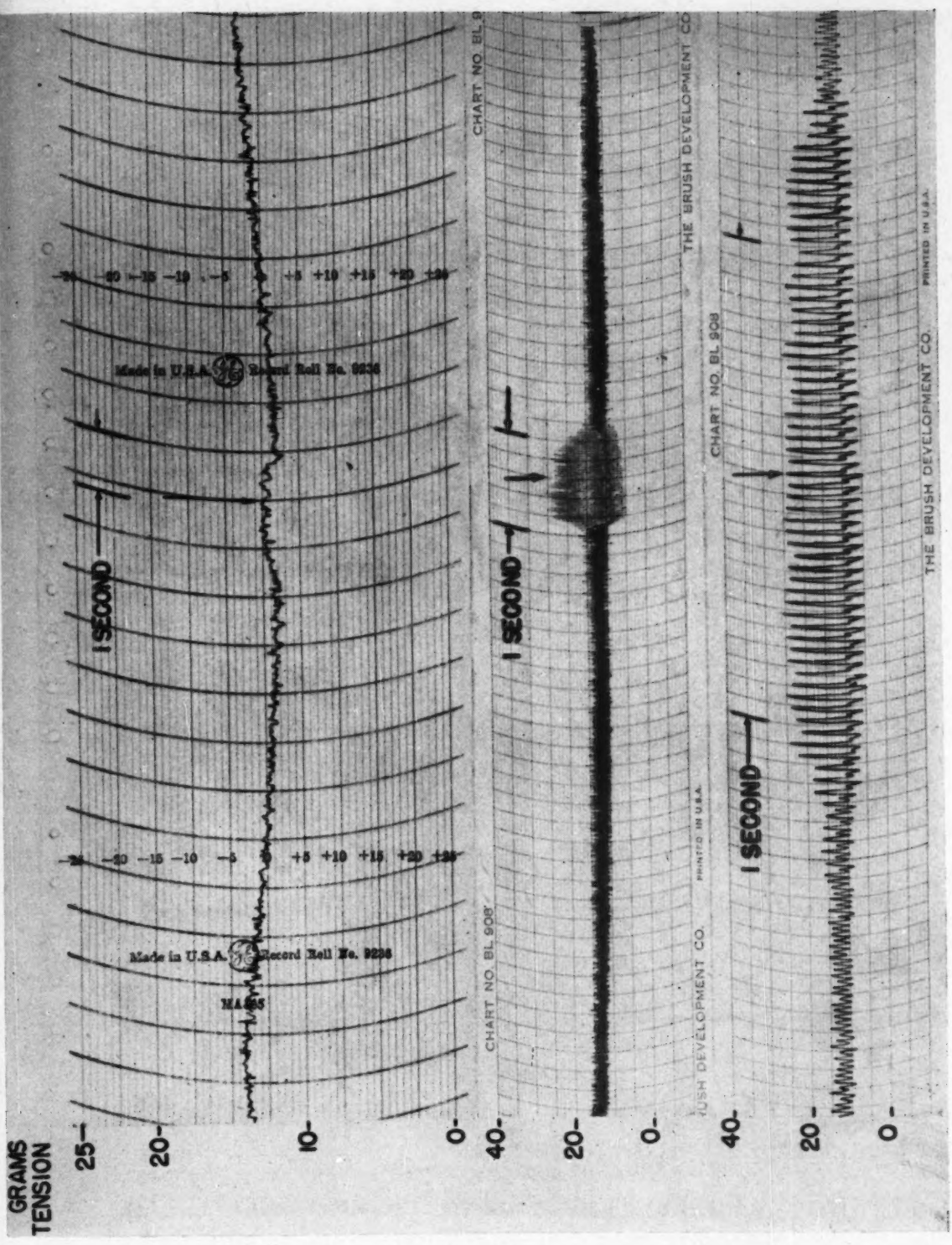

FIG. 8. *Tension variations near top of traverse; yarn running on ring spinning frame.*

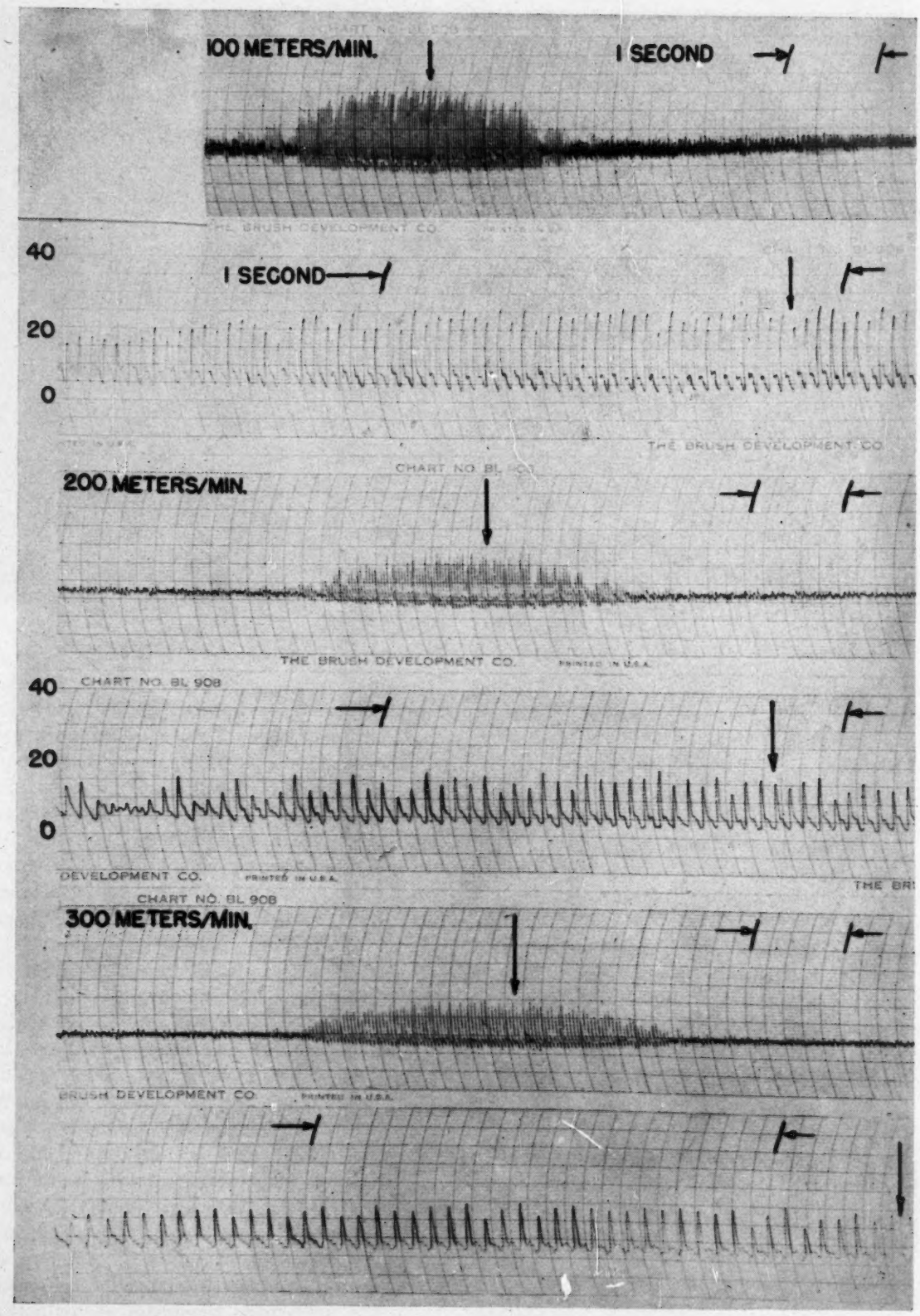

FIG. 9. *Tension variations near top of traverse. Various yarn feed rates.*

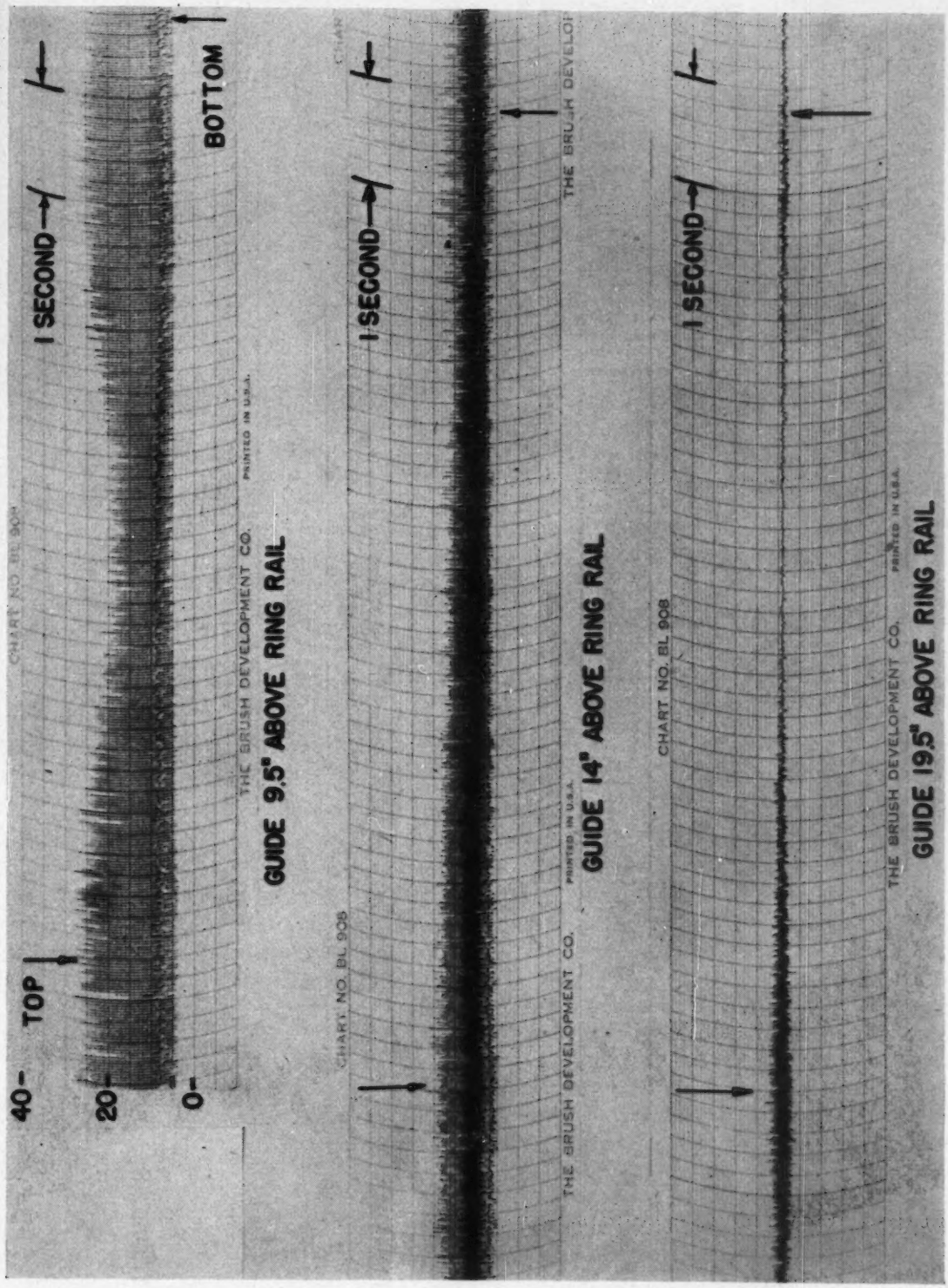

FIG. 10. *Tension variations during traverse. Fixed balloon lengths.*

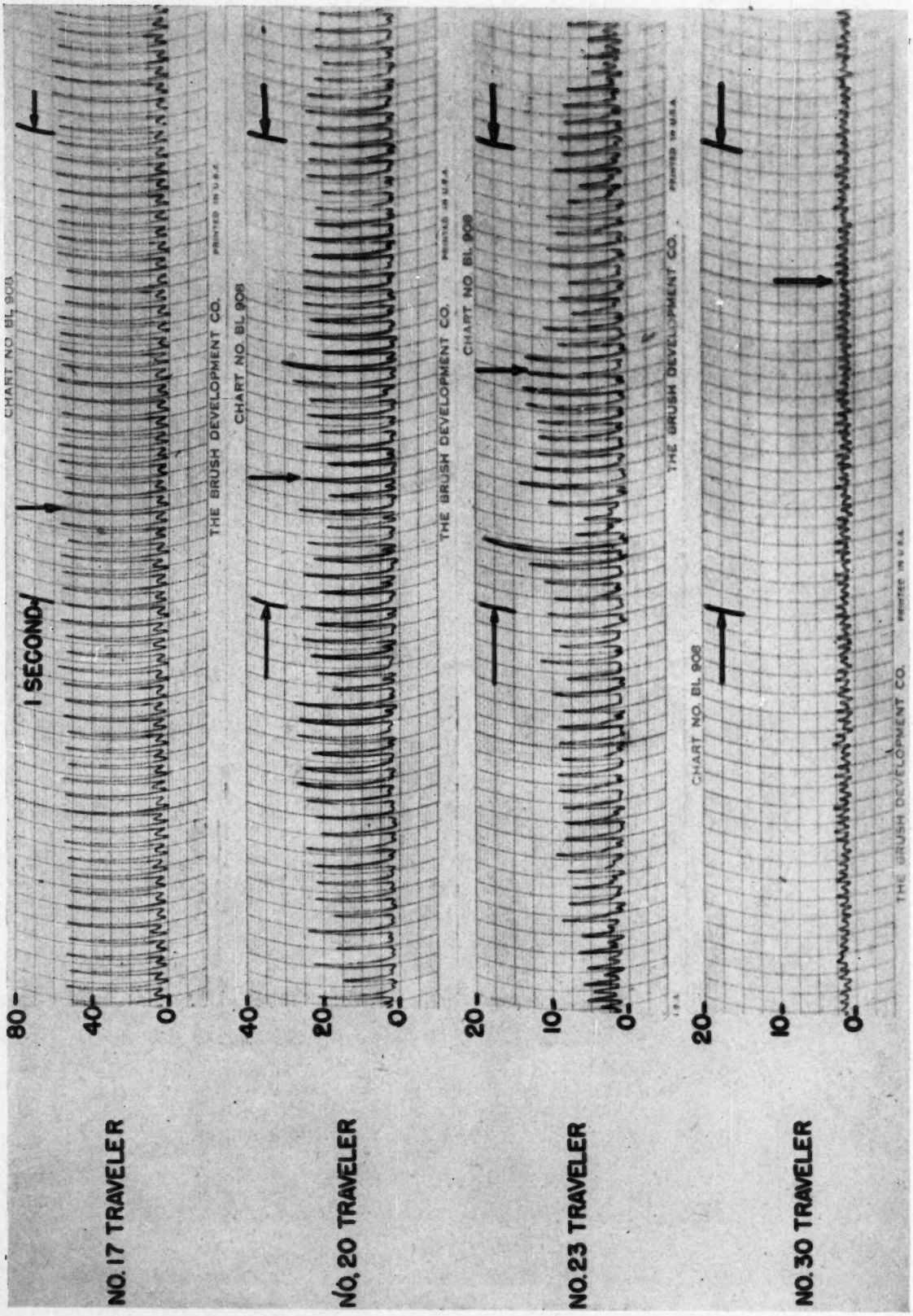

FIG. 11. *Tension variations near top of traverse. Various traveller weights.*

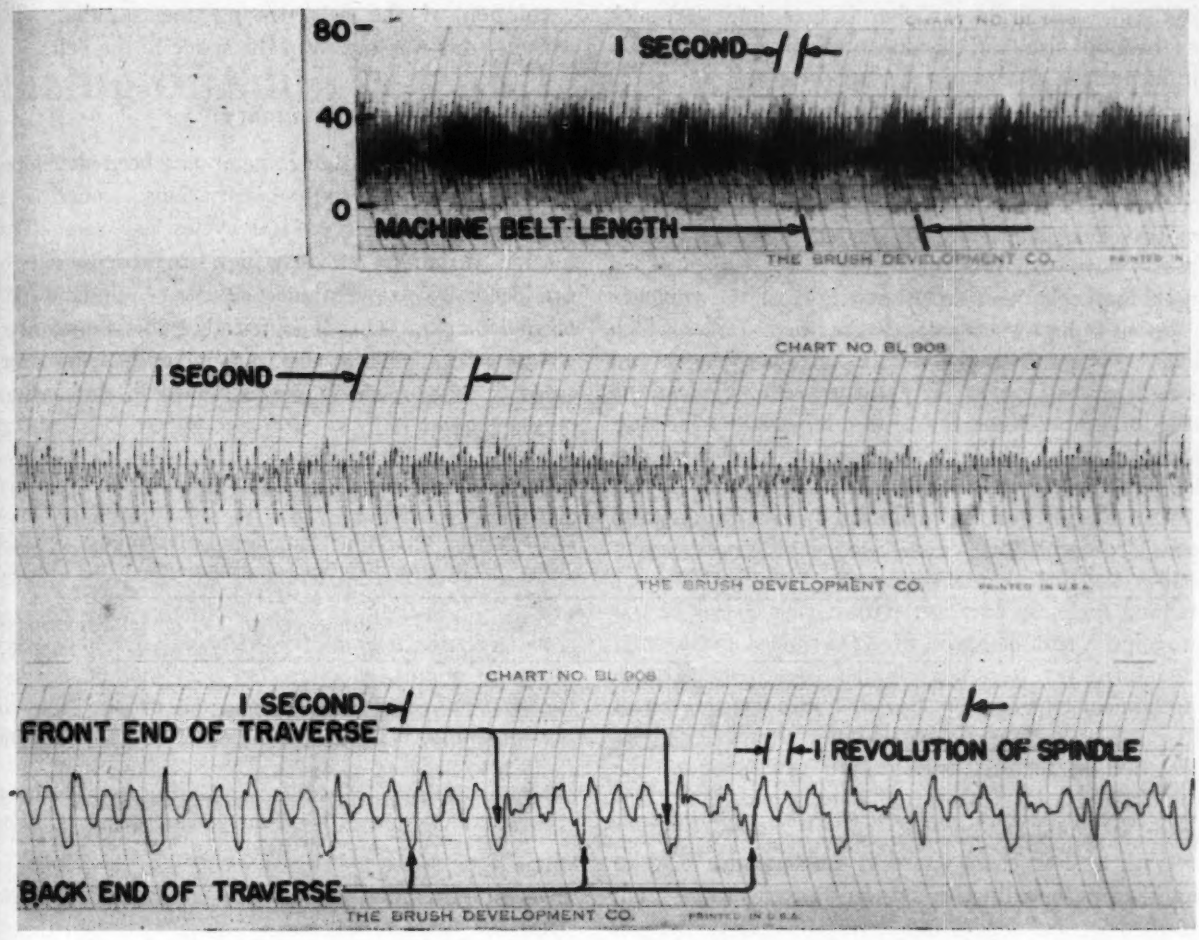

FIG. 12. *Tension variations during cheesing on Leesona Universal Winder.*

unit was modified so that the balloon guide was fixed to the traveller ring rail and rode with the ring rail as it traversed the bobbin. This made the length of the balloon constant over the entire cycle of the traverse. A balloon length equivalent to that at the top of the bobbin, where the largest tension variations occurred, was chosen first; then some intermediate length of balloon; and, finally, the longest length of balloon which had occurred during the previous experiment. Figure 10 shows charts for these three conditions, with a yarn speed of 100 m./min. and a spindle speed of 5240 r.p.m. Although there is still some variation from top to bottom of traverse, there is no doubt that the large variations are a function of the balloon length rather than of the position of the traverse on the bobbin.

TABLE I. FREQUENCY OF TENSION VARIATIONS AT VARIOUS YARN FEED RATES AND SPINDLE SPEEDS

Yarn feed rate (m./min.)	Spindle speed (r.p.m.)	Traveller speed (r.p.s.)	Tension variation (cycles/sec.)
50	5240	81.4	81.5
100	5240	75.3	75.5
200	5240	63.5	63.0
300	5240	51.6	51.5
50	5890	92.2	92.2
100	5890	86.2	86.6
200	5890	74.2	75.1
300	5890	62.5	65.0

The effect of traveller weight on the tension variations was studied by using various weight travellers, with other conditions kept constant. Figure 11 shows the results obtained with travellers varying from No. 17 (heavy) to No. 30 (light), with a yarn speed of 200 m./min. and a spindle speed of 4900 r.p.m. It appears from these results that the very light traveller

is the most desirable. Other factors, however, such as balloon size and hardness of the package, also influence the choice of traveller weight.

Two possible explanations may be advanced for the occurrence of the high peaks in tension at some point in the traverse stroke. One is that mechanical resonance of the balloon occurs with a certain weight of traveller, length of balloon, and average tension in the yarn. Another theory is that the weight of the yarn in the balloon and the weight of the traveller rotating at high speeds cause centrifugal forces which may act on the traveller with a certain length and weight of balloon to force the traveller against the ring rail and thus cause the tension to increase sharply [2]. Not enough evidence has been collected as yet to prove either theory.

The tensiometer has been used on other high-speed textile equipment with equal success. Figure 12 gives some results obtained on a Leesona Universal Winder making a cheese package, with a yarn speed of 100 m./min., a spindle speed of 1200 r.p.m., and a wind of 3 turns per traverse. Under these conditions, a mechanical tensiometer showed a uniform tension of 25 g. The high-speed tensiometer shows a definite variation in tension during each revolution of the spindle and a large variation at each end of the traverse, as would be expected.

With a slow chart speed it was possible also to note a difference in the tension pattern during each revolution of the belt driving the spindle. The cause of this was traced to the splice in the belt.

Summary

A recording yarn tensiometer has been developed which will record tension variations occurring at a rate of the order of 100 cycles per sec. That tension variations of such high frequencies can be produced by conventional textile equipment has been demonstrated. It is felt that this tensiometer will prove valuable in the study of yarn being processed by high-speed equipment used in the various textile operations.

Literature Cited

1. Breazaele, F., Apparatus for Determining the Coefficient of Friction of Running Yarn, TEXTILE RESEARCH JOURNAL **17**, 27–31 (1947).
2. Goderville, C., Influence of Traveller Friction on Regularity of Yarn, *La Industrie Textile* **67**, 101–3 (1950).
3. Morton, W. E., "An Introduction to the Study of Spinning," London, Longmans, Green and Company, 1937.
4. Nadir, M. T., "Thread Tension Transducer," Dumont Oscillographer, Dumont Laboratories, Clifton, N. J., July–Sept., 1949.

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Sizing of Spun Nylon Yarns with Various Agents

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INTEREST in the sizing of spun nylon yarns is a natural consequence of the fairly recent commercial development of such yarns and of the considerable work which has been done on the sizing of filament nylon.

Obviously, the sizing requirements of the two types of yarn are quite different; and, while satisfactory nylon filament sizes have been developed, no completely satisfactory size for spun nylon has been found. Three of the nylon filament sizes have been used to some extent on spun nylon yarns. Each of these—polyvinyl alcohol, polymethacrylic acid, and polyacrylic acid—has certain points in its favor for this use, but, at the same time, each has very definite drawbacks.

In the present investigation, an attempt was made to develop a better size formulation for spun nylon, both by testing other materials as sizing agents and by modifying the three above-mentioned agents that are already in use. It was discovered quite early that greater promise seemed to lie in the latter phase of the investigation. Therefore, most of the data reported herein comprise the preliminary results of a study along those lines. One such modified size formula was studied rather closely all the way through large-scale slashing and weaving operations.

Experimental Procedure and Results

Materials

The yarn used in all preliminary tests described herein was 12/1 spun nylon, 3-denier, 1½-in. staple, made by the Yarn Manufacturing Department of the Clemson College School of Textiles. For slashing and weaving tests, a similar 18/1 yarn from the same source was used.

Polyvinyl alcohol was used in the form of Nylon Size #10 from Atlas Powder Company, polymethacrylic acid as DuPont's Tyze 2, and polyacrylic acid as Orthocryl 25 from American Aniline & Extract Company. The sodium alginate was Keltex from Kelco Company. Dimethylol urea was used in the form of DuPont's Arboneeld. Stymer S, a Monsanto product, was the source of polystyrene.

Other trade-name products came from the following sources: Tween 20 and Avcosol 131, Atlas Powder Company; Twitchell Oil-7234 and Emery Oil E 670-R, Emery Industries, Inc.; Warco-A-170, Warwick Chemical Company; Victawet, Victor Chemical Works; Avitex R and Sodium CMC (carboxymethylcellulose), DuPont Company.

All other chemicals were used in the regular commercially available forms.

Test Sizing with Various Agents

In order to approximate conditions in the slasher, the various size mixtures were applied to test samples of yarn by the following procedure:

A small skein (approximately 0.5 g.) was thoroughly impregnated in the size bath held at a given temperature, then lifted from the bath, the excess size paste being removed as completely as possible by scraping between the fingers, and then unwound through squeeze rolls. The squeezed yarn was wound, spacing the loops as they were wound, on a sample board winder (or seriplane) in which the board had been replaced by two large test tubes fitted over the board-holding prongs. The yarn wound on this rack was dried in an oven at approximately 100°C and rewound into skein form.

The effectiveness of the sizing agent in resisting yarn abrasion was tested by threading the sized yarn on a Duplan Silk Cohesion Tester and noting its abrasion-resistance, expressed either as the relative appearance of the yarn after a given number of strokes on the machine or as the number of strokes necessary to produce an appreciable fuzzing of the yarn surface.

Breaking strengths were measured on the Scott Single Strand Tester. For comparison purposes, the unsized yarn showed a breaking strength of 1360 g.

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The various sizing agents thus evaluated were as follows:

Dimethylol Urea.—A 5% solution of Arboneeld was prepared by dissolving the solid in cold water. In this very fluid solution at room temperature the yarn took up 9.2% of size, but was quite fuzzy and very soft. There appeared to be little, if any, change in the properties of the yarn.

Polystyrene.—This material was dissolved according to the procedure outlined in Monsanto's Technical Bulletin T14 by adding Stymer S gradually, with vigorous mechanical stirring, to about two-thirds of the total amount of water, boiling for 30 min. with continued stirring, and adding cold water to give the final desired volume. The yarn was sized in the clear, slightly viscous solution at 60°–65°C. The yarn took up 6.1% of its weight of Stymer S from a 7% solution, and 10.3% from a 10% solution. Both samples of sized yarn showed a high degree of fuzzing after only 30 strokes on the Duplan Tester.

Starch.—A 5% aqueous dispersion of corn starch was heated to a gelatinous paste. Sizing in this paste at 65°C gave 8.2% take-up by the yarn, but the sample was quite soft and fuzzy. Breaking strength was 1274 g.

Repeating the process with a 20% starch paste gave 30.1% take-up and a somewhat stiffer, but still fuzzy, yarn having a breaking strength of 1332 g. and showing poor resistance to fuzzing after 30 strokes on the Duplan Tester.

Gluconic Acid.—This material was obtained as an approximately 50% aqueous solution; it was diluted to the desired concentration, with or without various additives, and applied to the yarn at 66°C (150°F). The amount of additive, when used, was sufficient in each case to give a quite viscous solution. All of the sized samples were somewhat stiff immediately after removal from the drying oven; but, due to hygroscopicity, they soon became soft, sticky, and clammy. Weighings and cohesion tests (using 30 strokes on the Duplan Tester) were made before this condition had the opportunity to develop. Results of several such size tests are shown in Table I.

Polyvinyl Alcohol.—Nylon Size #10 was dissolved by sifting 13% of the solid polymer into the appropriate volume of water at 60°C, stirring mechanically all the while. Stirring was continued after heat was removed and until the solution reached room temperature. It was quite fluid. A sample of yarn sized at room temperature and dried was found to have taken up 10.7% of its weight of size, and was fairly stiff but somewhat fuzzy. After 50 strokes on the Duplan Cohesion Tester the yarn was badly fuzzed, and considerable shedding was noted.

The residual solution used above was divided into two parts. To one part was added 2% of sodium alginate (Keltex), which was dispersed by stirring at 65°C. Yarn sized in this mixture at room temperature took up 39.5% of size (dry weight). It showed slightly less fuzzing after 150 strokes on the Duplan Tester than the previous sample did after 50 strokes. Shedding of the size during this test was appreciable.

To the other part of the original size mixture was added 6% of polymethacrylic acid (Tyze), which was dissolved by stirring and heating to 65°C. The very fluid solution, cooled down to room temperature, had a fairly stiff consistency. Yarn sized therein took up 54.7% of its weight of the size, but showed about the same degree of fuzzing after 95 strokes on the Duplan Tester as did the sample sized with polyvinyl alcohol and sodium alginate after 150 strokes. Both of these latter samples were stiff but had a somewhat waxy feel.

Polyacrylic Acid.—Water was added to Orthocryl 25 to reduce the solid content from 25% to 12%, giving a quite fluid solution. The size take-up by a sample sized at 57°C (135°F) was 12.6% of the yarn weight, giving a somewhat fuzzy yarn of good flexibility. Fifty strokes on the Duplan Tester caused considerable fuzziness, which increased gradually on further abrasion. After 300 strokes the yarn was badly fuzzed but still unbroken.

Another sample of yarn was sized with a mixture containing 12% of Orthocryl and 2% of Keltex. This solution was more viscous than that without Keltex, but was still reasonably fluid. Takeup was 14.6%, and the yarn was slightly stiffer than that sized without the alginate. Abrasion-resistance was

TABLE I. RESULTS OF SIZING SPUN NYLON YARN WITH GLUCONIC ACID PLUS ADDITIVES

% Gluconic acid	Additive	% Takeup	Duplan test
25	None	—	Poor
25	Bentonite	30.4	Good
25	Gum arabic	26.5	Poor
25	Glue	25.4	Fair
25	Sodium alginate	33.7	Good
5	5% Starch	11.0	Poor*

* This sample had unusually high breaking strength—above 1600 g.

better in this case, however, showing only moderate fuzzing after 300 strokes on the Duplan Tester.

A size mixture containing 6% of Orthocryl and 4% of Keltex had the consistency of stiffly whipped cream. There was no flow, but the mass was not stiff nor stringy. A sample of yarn took up 6.3% of its weight from this mixture. The sized yarn fuzzed badly and broke after 75 strokes on the Duplan Tester.

Polymethacrylic Acid with Added Plasticizers or Thickeners.—The general method of preparing these size formulations consisted of slowly sifting a weighed quantity of Tyze 2 into the correct volume of cold water while stirring vigorously, then adding the plasticizer or thickener, and heating the mixture to 66°C, with stirring, until all lumps were dispersed. The amount of thickener used in each case was sufficient to give a fairly viscous consistency. Duplan cohesion tests were run on all samples, using 30 strokes on the machine unless otherwise noted. Table II gives the results of all tests using these formulations.

TABLE II. TESTS OF SPUN NYLON YARN SIZED WITH POLYMETHACRYLIC ACID WITH ADDED PLASTICIZERS AND THICKENERS

% Polymethacrylic acid	Additive	Sizing temp. (°C)	% Takeup	Duplan test	Breaking strength (g.)
	Plasticizers				
6	3% Glycerol	90	7.6	Good	—
8	3% Glycerol	90	10.1	Good	—
8	6% Glycerol	66	17.1	Fair	860
8	6% Sorbitol	66	18.4	Poor	1475
5	25% Gluconic acid	66	51.5	Fair	1062
6	4% Gluconic acid	66	13.0	Poor	1195
8	4% Gluconic acid	66	20.0	Fair	1150
8	25% Gluconic acid	66	70.0	Fair	—
8	25% Gluconic acid + 10% Talc	66	97.6	Poor	—
	Thickeners				
8	None	66	56.1	Excellent*	—
5	Stymer S	66	18.2	Fair	1376
5	Starch	66	11.8	Poor	1168
3.3	Starch	66	6.9	Poor	1259
5	Bentonite	66	7.1	Poor	—
8	Glue†	66	99.8	Poor	—
8	Gum arabic	66	22.7	Poor	—
7.2	2.4% CMC	48	5.5	Poor	—
7.2	5% CMC	48	7.6	Poor	—
8	Keltex	66	34.0	Excellent‡	1347

* After 200 strokes on the machine. This size mixture was extremely stiff and rubbery.

† Incompatible with polymethacrylic acid at 66°C.

‡ After 100 strokes on the machine.

Polymethacrylic Acid and Sodium Alginate Under Varying Conditions

In general, the size mixtures were prepared by moistening the weighed quantity of Keltex with alcohol and dispersing in half the total amount of water to be used. The Tyze was dissolved in the rest of the water by vigorous stirring at room temperature, and then heating to 66°C. The resulting solution was mixed thoroughly with the Keltex dispersion, and the temperature was adjusted. The size was applied to the yarn as previously described. All sized samples were tested on the Duplan Cohesion Tester; the results are expressed as the number of strokes on the machine which were necessary to develop a light, but appreciable, fuzz on the yarns.

Results of these tests are summarized in Table III. Single-strand breaking strengths determined on selected sized samples were all of the order 1250–1280 g.

TABLE III. TESTS OF SPUN NYLON YARN SIZED WITH POLYMETHACRYLIC ACID AND SODIUM ALGINATE

% Polymethacrylic acid	% Sodium alginate	Sizing temp. (°C)	% Takeup	Solution consistency	Duplan test
0	4	66	4.0	Soft, no flow	44
2	3	90	2.8	Fluid	20
4	3	90	19.9	Viscous	200
4.8	2.4	90	8.9	Viscous	30
6	2	90	24.9	Stiff, stringy	125
6	3	90	24.7	Stiff, stringy	1000
6	3	80	15.7	Stiff, stringy	150
6	3	85	19.5	Stiff, stringy	250
6	3	85*	29.7	Stiffer, stringier	300
6	3	30†	33.1	Very stiff	300
6	3	85	35.0	‡	300
6	3	30**	25.5	Fairly stiff	300
6	3	66	14.5	Viscous, stringy	500
6	3	30	12.8	Viscous, stringy	200
6	3	48	12.7	Viscous	90
8	1	90	10.2	Stiff	50
8	2	90	26.3	Very stiff	300
8	3	50	27.5	Rubbery	300

* The size mixture used on this sample was the same as that used on the previously listed sample, but it was heated at 85°C for 15 min. longer to give a stiffer paste.

† This size paste was the one described above (*), which was cooled to room temperature before being applied to the yarn.

‡ For this sample the size paste described above (†) was heated at 85°C for 1 hr. longer. The dry film which formed on the surface of the paste was stirred, enough water was added to give a smooth consistency, and the sample was sized at 85°C.

** Same size paste as above, cooled to room temperature before being applied to the yarn.

Polymethacrylic Acid-Sodium Alginate Mixtures with Added Plasticizers

The same general procedure described above was followed for preparing mixtures containing 6% of Tyze and 3% of Keltex, except that before heating to the sizing temperature a measured amount of plasticizer was added to each mixture. Results are given in Table IV.

TABLE IV. TESTS OF SPUN NYLON YARN SIZED WITH POLYMETHACRYLIC ACID AND SODIUM ALGINATE WITH ADDED PLASTICIZERS

Plasticizer	Temp. (°C)	% Takeup	Solution consistency	Duplan test
3% Glycerol	90	20.0	Viscous	150
6% Glycerol	90	17.6	Viscous	150
2% Warco-A-170	80	20.0	More fluid	37
1% Victawet	30	14.3	Very fluid	30
2.5% Twitchell Oil-7234	66	12.0	Quite fluid	110
1.5% Emery Oil E670-R	66	18.0	Quite fluid	125

Desizing Spun Nylon Treated with Polymethacrylic Acid and Sodium Alginate

A sample of yarn sized at 90°C with 6% of polymethacrylic acid and 3% of sodium alginate was stirred gently in lukewarm water for 5 min., then rinsed in cold water, dried at 100°C, and weighed. The loss in weight from this process was 38.0%, compared with a takeup value in the sizing operation of 37.8%.

Loom Abrasion Test on Yarn Sized with Polymethacrylic Acid and Sodium Alginate

A skein of yarn was sized with 6% of Tyze and 3% of Keltex at 90°C. Approximately 125 ends of this sized yarn in 1-yd. lengths were tied into a 30/2 cotton warp on a Draper plain loom set up for a 3-harness twill with the let-off motion and the take-up motion disconnected. The loom was run in the same place with an empty shuttle for 30 min. at 160 picks per in., 63 ends per in. in 20 reed, 3 ends per dent. At the end of this time there appeared to be little, if any, fuzziness on the sized nylon. Nor did any appreciable fuzziness develop after 1 hr. under the same running conditions.

The test was repeated using the same yarn sized with 6% of Tyze and 3% of Keltex at 66°C. Again, after 30 min. of the test, the yarn showed little, if any, fuzziness.

Slashing of Spun Nylon Warp with Polymethacrylic Acid and Sodium Alginate

Thirty gallons of a size mixture containing 6% of Tyze and 3% of Keltex was prepared by dispersing each solid ingredient in half the total volume of water and then mixing the two. It was found that in bulk these concentrations of ingredients gave a mixture that was too thick to flow readily from the mixing kettle to the size box of the slasher. Consequently, water was added to the mixture to produce a workable consistency. The resulting concentration was 4.0% with respect to polymethacrylic acid, and 1.8% with respect to sodium alginate.

A 150-yd. warp of 6 section beams, 512 ends to the beam, prepared from 18/1 spun nylon yarn (3-denier, 1½-in. staple) was slashed with this mixture on a 7-can Cocker rayon slasher, at a size-box temperature of 150°F (66°C). Drying-can temperatures were 160°, 180°, 190°, 190°, 200°, 180°, 180°F.

The warp so slashed was pliable but somewhat harsh, and had very little surface fuzz. Only slight shedding was noted at the split rods.

Desizing a sample of the warp with warm water showed the takeup of size to have been 10.4%.

Weaving of Spun Nylon Warp Slashed with Polymethacrylic Acid and Sodium Alginate

The warp slashed as described above was woven with 18/1 spun nylon filling on a Draper X-D model loom with dobby of 20-harness capacity, set up for a 3/2/1/2 twill weave with 40 picks per in., 74 ends per in. in 22½ reed, 3 ends per dent. The results of this weaving operation were quite satisfactory. The yarn was pliable in weaving, practically no flaking occurred, and there was no noticeable chafing or beading. The fabric was easily desized by the procedure used on the sized yarn.

Summary and Discussion

In addition to the general requirements of any size mixture, the most important criterion of a satisfactory size for spun nylon is that it hold down the ends of the staple fibers in the yarn, which otherwise tend to work to the surface during the abrasive action of the weaving process, causing fuzzing of the yarn surface and consequent tangling of the warp ends. From this standpoint, starch, polystyrene, and dimethylol urea were completely ineffective as

sizing agents. Gluconic acid offered some promise, but was too hygroscopic for practical use. Thickeners used with gluconic acid decreased hygroscopicity somewhat, but also reduced anti-fuzzing action.

More effective as anti-fuzzing agents were the materials which have been used successfully to size filament nylon—polymethacrylic acid, polyacrylic acid, and polyvinyl alcohol, in that order. Each of these had certain drawbacks, however. Polymethacrylic acid, although noncorrosive in the dry state, is quite acidic in solution, necessitating the use of stainless-steel equipment. Without a plasticizer it gave very stiff yarns; with a plasticizer it lost much of its abrasion-resistance. Polyacrylic acid is also highly acidic in solution, and was found to be somewhat inferior to polymethacrylic acid as an anti-fuzzing agent. Polyvinyl alcohol was less effective, and, in addition, showed a pronounced degree of shedding from the yarn.

Of the various materials used to increase the cohesion of polymethacrylic acid to the yarn, only sodium alginate showed any degree of effectiveness; and it caused marked improvement not only in the case of polymethacrylic acid but also, to a lesser degree, with polyvinyl alcohol and polyacrylic acid.

The effect of the alginate is interesting since, in itself, sodium alginate is not a good size for the spun yarn. The incorporation of alginate into the other size mixtures increased the viscosity in each case, and, in most cases, it increased the takeup of the size by the yarn. In the more extensive work with polymethacrylic acid, both with sodium alginate and without, the higher the temperature and the longer the heating time to which the size mixture was subjected, the stiffer it became and, in general, the more effective as to size takeup and abrasion-resistance on the yarn. No unequivocal relationship between take-up and abrasion-resistance could be established, however.

Since the Duplan method of determining abrasion-resistance gave only relative measurements and could not be used to set up a standard minimum anti-fuzz value, the results obtained by tying samples of polymethacrylic acid-sodium alginate-sized yarn into a warp on a loom and running the loom at a stationary position with an empty shuttle were especially revealing. It was calculated that the maximum length of time in which the sized warp ends would be subjected to abrasive action on this loom was 7 min. Since the two samples of sized yarn so tested showed no signs of fuzzing in the respective 30 and 60 min. of the tests, it was obvious that size mixtures of lower actual anti-fuzz efficiency would be perfectly satisfactory for practical use. This was important since the two samples tested had been sized with mixtures which were so extremely viscous and stringy as to be entirely impractical for use in a slasher. This indicated that the lower Duplan values obtained from yarns sized at lower temperatures and with plasticized polymethacrylic acid-sodium alginate mixtures (which had much lower viscosities) might still be within the range of acceptable abrasion-resistance for practical usage.

Further confirmation of this point was offered by the perfectly satisfactory weaving results obtained from a warp which was actually sized in a slasher with a mixture containing only two-thirds the concentration of active ingredients which had produced the sized sample of maximum Duplan value. Added significance may be imparted to the results of this weaving test since it used a closely woven fabric construction similar to a good-quality worsted suiting. The warp yarns were, therefore, subjected to more drastic chafing action than would have been true in the case of a looser construction.

This slashed warp had a slight tendency toward harshness, and at concentrations higher than those discussed above the size mixture showed some tendency toward stringiness and sticking to the rubber squeeze rolls of the slasher. However, at the concentration reported (4.0% of polymethacrylic acid and 1.8% of sodium alginate) this tendency was not apparent. The use of plasticizers in the mixture would probably decrease the stickiness as well as the harshness of the yarn, while allowing the practical use of higher concentrations of polymethacrylic acid and sodium alginate if desired. Further slasher and weaving tests should be made in order to work out the optimum size formulas for these operations.

These results might be interpreted further as suggesting that the abrasion-resistance imparted to spun nylon by sodium alginate-thickened polyvinyl alcohol and polyacrylic acid, while inferior to that shown by polymethacrylic acid, might be sufficient for practical purposes. These two mixtures warrant further investigation.

An important factor in the use of polymethacrylic acid-sodium alginate size mixtures is their ease of removal from the yarn or fabric, a short treatment

with lukewarm water followed by a cold-water rinse being sufficient to completely remove the size, even when it is present on the yarn in large amounts.

It is claimed by some that a drawback to the use of polymethacrylic acid on filament nylon is the incompatibility of the size material with the spinning oils used on the fiber. Simple tests showed that three spinning oils—Avcosol 131; Tween 20, which is generally used on low-denier nylon staple; and Avitex R, which is used on higher deniers—in aqueous solution are all incompatible with an aqueous solution of polymethacrylic acid, forming a precipitate. With a dilute aqueous solution of polymethacrylic acid and sodium alginate, solutions of Avitex and Avocosol still gave precipitates, but Tween 20 did not. However, in view of the small amount of oil on the yarn (1% to 2%) and the viscosity of the polymethacrylic acid-alginate size mixtures, it is considered unlikely that enough oil would be removed from the yarn under ordinary sizing conditions to cause any difficulty.

It should be mentioned that in the case of nylon yarns, which have high inherent strength, the effect of sizing on breaking strength is relatively unimportant. It was found in this investigation that sizing with polymethacrylic acid and sodium alginate did not increase the breaking strength of the yarn, the inherent strength of the yarn being greater than the bonding strength of the size. Neither, however, did sizing have any significant adverse effect on the strength.

Conclusions

(1) Of the several potential sizing agents for spun nylon which have been evaluated, the most promising appear to be polymethacrylic acid, polyacrylic acid, and polyvinyl alcohol.

(2) The sizing efficiency of all these materials is increased by the addition of 1%–3% of sodium alginate to the size mixture.

(3) A size mixture containing 4.0% of polymethacrylic acid and 1.8% of sodium alginate has been used with good results in the slashing and weaving of a 18/1 spun nylon warp.

(4) Further and more complete investigation into the use of alginate-containing spun nylon size mixtures should be carried out.

Acknowledgment

Appreciation is expressed to the various industrial companies mentioned herein for their interest in this work, as expressed in the form of samples and information. Thanks are due to the various members of the Department of Weaving and Designing of the Clemson College School of Textiles who lent their assistance in the slashing and weaving operations described, and particularly to Mr. A. E. McKenna, Head of the Department of Weaving and Designing, and to Mr. Joseph Lindsay, Jr., Head of the Department of Textile Chemistry and Dyeing, for help in many ways.

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1951 Annual Meeting, T.R.I.

Technical Session

Because of the widespread interest in the papers delivered at the 1951 Annual Meeting of Textile Research Institute (Hotel Statler, New York City), the papers are being published in full in TEXTILE RESEARCH JOURNAL. *In the following pages of this issue are the papers given at the Technical Session on Thursday, November 8, 1951. Those given at the General Session on Friday appeared in the January issue.*

An Analysis of the Consumer Quality Characteristics of Cotton: Implications for Research

George S. Buck, Jr.*

TEXTILE RESEARCH may be considered to have three broad areas of application. These are: (1) producing the fiber; (2) adapting the fiber to mill processing; and (3) developing in the fiber and product those qualities which the consumer desires. Important as are the first two areas of research, they must nevertheless be somewhat subordinated to the third in this age of growing interfiber competition for markets. Specialized requirements in textile end-uses demand specialized properties in fibers and fabrics. Other factors being equal, the fiber having the most satisfactory functional performance in a particular application will capture that market.

Cotton serves hundreds of different end-uses well because of its versatility. Those in the cotton industry, however, recognize the need for the fiber and its products to meet increasingly specialized requirements in many applications. These extra performance demands result in large measure from the development of new synthetic materials which have unusually high performance values in some characteristics.

To be useful as a guide for research, an analysis of end-use performance criteria should include statistical data that give some idea of the relative importance of the desired quality improvements. Market research has proven a most useful tool not only in defining the objectives toward which cotton research must move, but also in weighting those objectives with figures measuring the size of the uses in which a given quality characteristic is important (in terms of bales of cotton or yards of cloth) and in providing information on the cost and availability of competitive materials having the desired properties. Thus, the first steps can be taken toward the selection of the more critical problems and the more promising opportunities.

This approach is not simply a matter of trying to insure the success of a project before it is undertaken, although that may also be involved. It is instead a recognition of the fact that research funds are all too frequently limited. Therefore, it makes scientific sense to use such funds with maximum efficiency.

The term "quality" used herein is meant to imply functional performance rather than relative excellence. Figure 1 lists 31 qualities or quality characteristics that market research studies have shown to be functionally important in one or more of 117 major end-uses of cotton yarns and fabrics. These are further weighted in the following manner. First, the qualities important in each of the 117 end-uses are recorded. Then only those qualities in which cotton

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CONSUMERS OF COTTON YARNS AND FABRICS WANT HIGHER VALUES IN THESE QUALITY CHARACTERISTICS

1. DRAPE
2. FAST DYES
3. VIVID PRINTS
4. LUSTER
5. SOFT COLORS
6. PERMANENT CRISPNESS
7. SHEERNESS
8. TENSILE STRENGTH
9. FLEX LIFE
10. QUICK DRYING
11. ABSORBENCY
12. COOLNESS
13. WARMTH
14. RESILIENCY
15. ELASTICITY
16. DIMENSIONAL STABILITY
17. INSULATING PROPERTIES
18. ADHESIVE QUALITIES

RESISTANCE TO:
19. WRINKLES
20. SOIL
21. STAINS
22. ABRASION
23. WEATHER
24. MILDEW
25. CHEMICALS
26. CLINGING
27. WATER
28. FIRE
29. WIND
30. HEAT
31. LINTING

FIG. 1

needs improvement for a particular use are tabulated. Now the total yards or pounds of cotton in those uses in which each quality appears produces a ranking of consumer requirements in terms of quality characteristics.

As an illustration, market investigators find that higher absorbency is desirable in only 9 of the 117 classified uses. Men's socks, babies' diapers, children's underpanties, and dish towels are examples of such uses. The total of the 9 uses consumes about 200,000 bales of cotton annually.

Higher luster, on the other hand, is found to be desirable in 46 of the 117 uses. It is important in sport shirts, dress socks, many items of women's wear, luggage lining, and other uses. The total of these 46 uses amounts to over 2,000,000 bales of cotton. Thus, from the standpoint of weighting the requirements of the ultimate consumer, an increase in the luster of cotton is considerably more desirable than an increase in cotton's already favorable absorbency.

Having determined the objectives of research in this field, and the relative importance of quality improvements in terms of needs and opportunities (and this judgment gives due weight to the cost and availability of competitive materials), one can study certain of the quality characteristics more carefully. This next step might be considered a technical analysis of market research information. In the cases of wrinkle-resistance and luster, fairly extensive studies of this nature have been completed. Other studies of a similar type are in progress. In greatly condensed form, some of the findings of these analyses

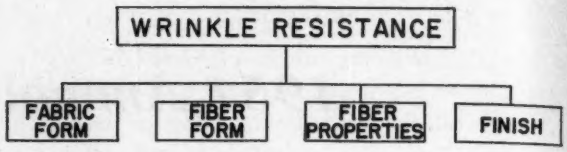

FIG. 2

can be illustrated, as well as the conclusions that have been drawn from them.

The selection of 4 of the 31 quality characteristics will illustrate the method; these are wrinkle-resistance, drape, abrasion-resistance, and warmth. None of these will be considered in any great detail. The information is, in fact, far from complete on each one, at least in the form presented here; but it is believed that some valid conclusions can be drawn from such a study even in this simplified form.

In Figure 2 factors contributing to wrinkle-resistance in textile fabrics are classified under yarn or fabric form (or form factor), fiber properties, and finish. It will be obvious that this simplified breakdown of a fabric property must necessarily be incomplete—may in fact be misleading unless used properly. For example, thick fabrics and coarse fibers add resistance to wrinkling and mussing forces, but crease-recovery may be difficult here once the elastic limit of the fiber has been exceeded. Similarly, soft yarns and loose weaves, as well as interfiber lubricants, permit shifting and deformation of the structure to reduce the strain in the fiber itself. But if the fiber, unlike cotton, were perfectly elastic, a tighter structure would have a more lively recovery because the fiber's power of recovery would be more readily transferred to the fabric.

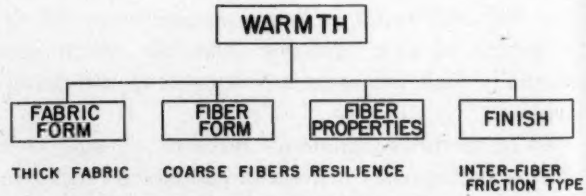

FIG. 3

The greatest significance of a chart of this type is what it shows in relation to similar charts on other physical properties. For warmth, illustrated in Figure 3, a thick fabric, soft yarns, and raised fibers are listed as favorable factors, just as in wrinkle-resistance. But here the reasons are different; these three factors now contribute to the effectiveness of an insulating layer of small air spaces. Coarse fibers are also listed as a favorable characteristic, partly because they resist compression better than fine fibers, but also because they resist twisting forces when spun into yarns, and the consequent tendency to untwist helps retain loftiness in the yarn. Crimp also helps maintain loftiness, and long fibers give more nap or pile and still leave enough fiber to anchor to the basic structure. Of course, an advantage of shorter fibers is that they provide more fiber ends to produce fuzzier yarns and a warmer "feel."

The finish type indicated in Figure 3 is just the opposite type from that shown in Figure 2. For wrinkle-resistance a lubricant type permits fiber shifting and a reduction of fiber strain; for warmth a finish that tends to anchor fibers together at various points helps prevent shifting to a more compressed form. The maintenance of a network of fiber arches results in the lofty yarn structure desired for warmth.

There is no conflict in the fiber physical properties suitable for warmth and for wrinkle-resistance. Resilience, as everyone knowns, is important to each. The ability of the fiber to recover from imposed strains smoothes out wrinkles and maintains loftiness. Note that wool has the desirable physical properties for both of these consumer characteristics.

Abrasion-resistance (Figure 4) shows similarities to the properties listed on both the wrinkle-resistance and warmth charts. Thick fabrics, where possible, are desirable because there will be more material to be worn away. Coarse fibers are indicated because abrasive forces are generally considered to be of small magnitude, and with a coarse-fiber strains should not reach the elastic limit so readily. Firm yarns and a firm weave are also desired. For certain types of abrasion, with relatively inelastic fibers, the freedom of fiber movement in softer structures may be useful. But if the term "abrasion-resistance" also connotes service wear—and in the market research sense it does—tighter yarns and a firm weave help to prevent snagging and pulling out of fibers.

The fiber physical properties again show resilience to be important. The reason for this is fairly obvious: The resilient fiber can absorb energy again and again as the abrasion cycle is repeated, while the work done on a nonresilient fiber is cumulative and reaches the fiber's limit sooner. But this resilience should not be confused with elasticity—the glass fiber is elastic and strong, but because of its limited extensibility it can absorb little work. The ability to absorb work, as measured by the area under the stress-strain curve, is regarded by many as almost a measure of abrasion-resistance. Possibly, the correct procedure isn't quite that simple, but such a measure is certainly related in an important manner to abrasion-resistance.

The fourth example of a consumer quality characteristic, drape, has its points of similarity and dissimilarity to the other three qualities (see Figure 5). Soft yarns, loose weave, and an irregular weave (other than plain) make for easier bending and folding. Fine fibers and flat fibers are also bent more easily than coarse fibers and round fibers. The interfiber lubricant type of finish aids fiber and yarn shifting; various commercial "softeners" are finishes of this type.

Resilience probably can be effective over a fairly short range in this case, in contrast to the type required for wrinkle-resistance and warmth, since the forces involved in drape are small. A low (initial) modulus should mean a fiber and fabric compliant with those low forces.

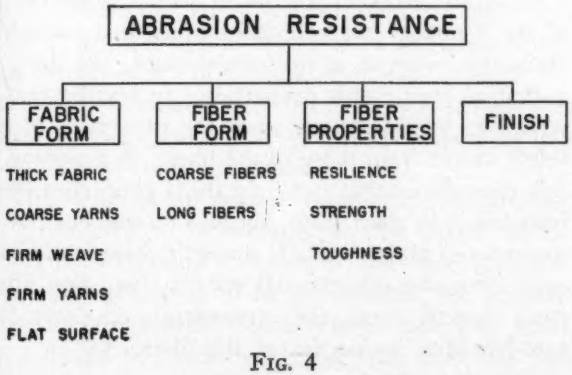

FIG. 4

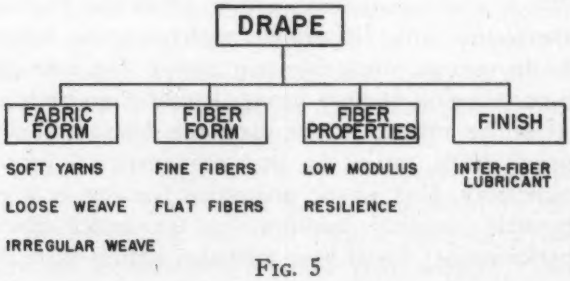

FIG. 5

Drape is an example of a fabric property that is understood by merchants, buyers, and retailers of fabrics, but which may be somewhat more difficult to describe in technical terms. Conflicts may arise in an attempt to outline its components on a chart. In such a case a detailed study of the property in the laboratory may be needed. With both luster and drape, such studies are in progress, and whether or not they produce the desired functional improvements, they certainly should result in a better understanding of the basic factors which contribute to those qualities in cotton.

Figures 2 to 5 represent in chart form only 4 of the 31 consumer qualities listed in Figure 1. By using much the same technique for each of the other properties, a grouping can be made which permits the attack of several objectives at once. Such a grouping would list the following quality characteristics under chemical properties: Soil- and stain-resistance, fast dyes, fire-, weather-, mildew-, and heat-resistance, water-repellency, and adhesive properties. In the same way the 6 or 8 properties strongly affected by fiber morphology (fiber shape) are segregated, as are those influenced by yarn and fabric construction. A fourth group, to be treated below in more detail, includes those properties which derive principally from fiber elastic properties, modified, of course, by yarn and fabric construction.

The grouping of objectives on the basis of their technical relationship provides an additional weighting of desirable lines of research. The importance of this should be obvious. If a certain research approach directly relates to 4 or 5 of the desired properties, it may become more valuable than another which affects only one quality characteristic, even though the latter, of itself, may be very desirable. Grouping also puts the objectives in better balance from a technical standpoint, so that sometimes general conclusions pointing to large areas of research may be drawn.

As this work is extended and refined, it is hoped that a cross-classification can be developed whereby the factors which determine quality can be related to the uses in which they best apply. For example, a combination of fiber morphological characteristics would be related to the yarn and fabric end-uses most ideally served by that combination. In the same way, fiber elastic properties (having in mind possible chemical modifications for better elastic performance) would have tabulated against them the applications requiring each type of elastic behavior. Obviously, this type of classification, if successfully completed, could be tremendously helpful in measuring the demand (or need) for various cotton fiber types, whether now commercially available or still in the experimental stages.

The fact that a complete analysis of fundamental qualities and its sequel, the relationship of fundamental fiber and fabrics characteristics to end-uses, require considerable study and research should not discourage one from drawing conclusions from preliminary studies. These screenings may be incomplete in some particulars, and may in some details be modified by more careful studies, but in general they will point in the right directions.

From such groupings of consumer qualities the conclusion is inescapable that the full potential in designing fabrics specifically for some of the desired consumer qualities is not being realized. Perhaps there is an overemphasis on strength and spinning performance, or too high a degree of competition in producing the so-called "standard" fabrics. Market research studies demonstrate a consumer demand that should mean good business for the enterprising mill designing and promoting cottons that, for example, drape better, wrinkle less, or have extra warmth.

Several broad lines of research are suggested by the quality summaries. First, research is needed on fabric geometry, with particular reference to the influence of construction variables on certain consumer qualities. Good work is being done in this field, largely with the synthetics; studies on cotton might well open the eyes of manufacturers to the value of specially designed cotton fabrics. Additional research is needed to relate available cotton fiber types to the uses and qualities for which they are best suited.

Certainly, however, the factor which influenced all of the 4 quality characteristics illustrated—wrinkle-resistance, warmth, abrasion-resistance, and drape—is that of fiber elastic properties. In textiles that is what one would expect, since the properties of the fabric derive from those of the fiber. A grouping in this case shows that not only the 4 properties illustrated here in chart form, but also 6 others of the 31 consumer qualities—tensile strength, sheerness, resilience, elasticity, dimensional stability, and flex life—stem directly from the stress-strain-time-temperature-humidity properties of the fiber.

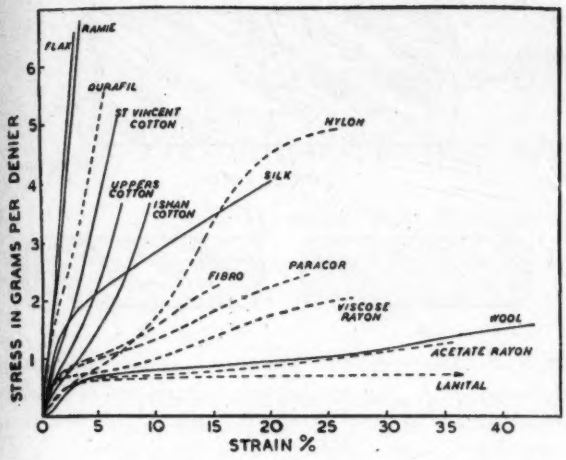

FIG. 6. *Load-elongation curves for natural and synthetic fibers.*

The complete stress-strain curves for textile fibers, including the curve for recovery from strain, can provide much information on the type of elastic performance a fiber should possess to result in certain fabric properties. Something, however, can be accomplished in a preliminary way, using analogy as a convenient tool, with the simple load-elongation curves of a number of fibers whose fabric characteristics are fairly well known. Figure 6 (from Meredith, *J. Textile Inst.* **36**, T107 (1945)) shows on a comparative basis the load-elongation curves of a number of natural and synthetic fibers. In using these for purposes of analogy, of course, some knowledge of the fibers' properties of strain recovery is employed, even though that part of the curve is not shown.

In Figure 6, a glance at the curves for fibers that have low resistance to wrinkling—flax, ramie, cotton, and viscose rayon—indicates that wrinkle-resistance does not stem from strength, nor from extensibility, nor from initial stiffness (Young's modulus). Fibers that resist wrinkles better in fabrics—acetate rayon, silk, and wool—show the same thing. But further study of the characteristics of these latter fibers in recovering from strains indicates that their superiority in that respect is the important factor in their wrinkle-resistance.

The range of our present commercial cottons may be considered to be bounded by the curve for St. Vincent Sea Island cotton and that for Ishan cotton. If a cotton is to be cross-linked for wrinkle-resistance, one with characteristics near the Ishan type should be much more responsive than one with the high modulus and more limited extensibility of the Sea Island type. That is because the cross-linking shortens the already limited cotton extensibility. In a "stretchy" cotton a more valuable, longer range type of resilience should result from cross-linking. Experimental cottons still more extensible than the Ishan type might well be investigated for receptivity to crease-resistant finishes, and further chemical work on crease-resistance should be in the direction of increasing cotton's extensibility before resin cross-linking.

The inferences on warmth, drawn in part from Figure 6, are much the same as those on wrinkle-resistance. If cotton is to be given the resilience of wool, the fiber toward which one instinctively looks in considering warmth, the cotton should preferably have more extensibility if cross-linking is to provide resiliency without harshness. Of course the interplay of fiber properties cannot be passed over too lightly. A fiber less crimpy than wool, having different frictional characteristics of the surface and less torsional rigidity, might well require a different stress-strain behavior and perhaps a higher modulus than is found in wool. However, it is extremely unlikely that cotton could be made as stretchy as wool, and there seems to be little doubt that a longer range elasticity than is now obtained from resin treatment and similar methods would be desirable for warmth.

There is much experimental evidence which points to fiber toughness, as measured by the area under the stress-strain curve, as being the fiber elastic property most directly related to abrasion-resistance. Nylon, among the fibers shown, typifies high abrasion-resistance. It can be seen that fibers having relatively poor abrasion-resistance can be either strong and nonstretchy like flax and ramie, or weak and stretchy like acetate and Lanital. Cotton's position is favorable, but would be improved if the curve inclined more to the right—in other words, if the fiber were more extensible. Resilience is important in abrasion-resistance, since the resilient fiber recovers from the cyclical strains imposed in abrasion. Since treatments that make cotton more resilient also decrease its extensibility and ability to absorb energy, the fiber should be as stretchy as possible before finishes of that type are applied.

Finally, for drape, a new set of values will rate the fibers. The rayons and wool, as opposed to less drapy fibers like flax and ramie, point to a low (initial) modulus as a desirable characteristic. This

seems reasonable, since draping forces should certainly be low. This again illustrates the desirability of a more stretchy cotton—one having a lower modulus.

It will be noted that fiber behavior has been dealt with rather lightly in reviewing the 4 characteristics —wrinkle-resistance, drape, abrasion-resistance, and warmth. This is deliberate. At this stage it is not necessary to explain why nylon should not resist wrinkles well, nor is it profitable to speculate on the relative drape of high-modulus silk and low-modulus nylon. The whole subject of fiber mechanics is extremely important in itself and deserves the support of all forward-looking textile men. But by its nature it cannot be considered superficially, particularly in those many phases which are presently receiving intensive study. Fortunately, cotton's needs are made sufficiently clear by the preliminary screening so that some satisfactory conclusions may be drawn.

An increase in extensibility and an improvement in its recovery from extension will be a major contribution to cotton's performance in the characteristics of wrinkle-resistance, drape, abrasion-resistance, and warmth. On the basis of this statement, attention can be directed toward the methods by which the objective may be reached.

If the highly crystalline structure of the cotton fiber, with its straight-chain inextensible molecules, were lined up perfectly with the fiber axis, gaining increased extensibility would indeed be a difficult task. Luckily this is not the case, for the internal structure, although highly organized, seems to form a spiralling, almost springlike, configuration in the cotton fiber. In undried cottons, removed from the boll before the crystal structure has had a chance to form, tension in the fiber results in considerable extension and in a realignment of the apparent molecular orientation to a position more nearly coincidental with the fiber axis (see Figure 7).

Even after the crystal structure is formed in the well dried, mature cotton fiber, there is an opportunity to loosen the structure and develop greater extensibility. Mercerizing without tension does this to a considerable extent, but still greater effects are achieved in the solvent-exchange type of decrystallizing treatment, which is under development at the Southern Regional Research Laboratory of the U. S. Department of Agriculture. It will be extremely interesting to watch the developments of the next few years in cottons having radically modified elastic properties. Even today there is one new commercially available finish for cotton which may be an indication of progress along this line.

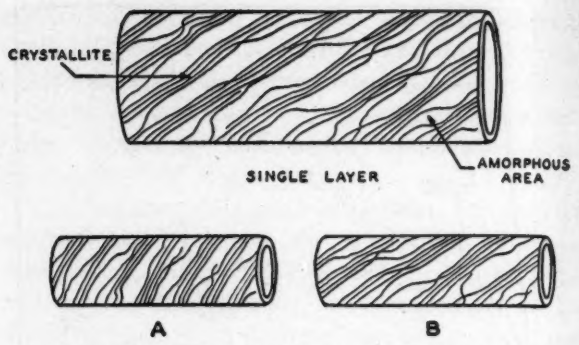

FIG. 7. *The crystallite orientation in undried cottons illustrated in* A *changes toward that shown in* B *upon stretching. Wide-angle cottons of the* A *type are more extensible in general than those of the* B *type.*

In addition to chemical methods for increasing the extensibility of the cotton fiber, careful attention should be given to opportunities in the selection and breeding of more stretchy cotton varieties. Several varieties of experimental cottons are known to be considerably more extensible than the most stretchy of the commercial types. While the plasticity of these cottons, together with their lower strength, limited the interest in their usefulness in the past, chemical methods for overcoming plasticity should warrant a re-examination of such extensible, wide-angle cottons.

Actually, one of the big needs in cotton research is for all who work with cotton to make some mental adjustment—to regard cotton not as a fiber with fixed properties, limited to the conventional uses it serves so well. Instead, cotton should be considered a basic fiber offering great possibilities for additional specialized uses through physical and chemical modifications accomplished by research.

The author wishes to emphasize that the subject reported here is part of a more extensive study intended to relate consumer needs in cotton textiles to fields of research. The methods of analyzing market research findings and of defining technical research objectives on the basis of those findings undoubtedly need much refinement. Suggestions for improving this work are welcomed.

Properties of Apparel Wools

I. Preliminary Report on Worsted Processing Trials*

Werner von Bergen† and James H. Wakelin‡

Contribution of the Forstmann Woolen Company, Passaic, New Jersey, and Textile Research Institute, Princeton, New Jersey

Abstract

Studies have been made on the physical and chemical properties and worsted processing characteristics of two fine and two medium wools. Each of the wools studied was found to have essentially the same basic physical properties when reduced to the same cross-sectional area and to have the same chemical composition. The average diameter or fineness of a wool lot is the most important characteristic of the wool insofar as the efficiency of manufacturing operations is concerned. By aging the wool at 70°F and 70% R.H., an improvement in spinning and weaving was observed. Steaming the wool in the form of roving was found to have the same beneficial effect on spinning as aging under normal room temperature conditions. The aging process appears to be related to the relaxation of those stresses to which the fibers are subjected in previous processing operations.

I—Introduction

Textile Research Institute and the Forstmann Woolen Company have cooperated in the Wool Research Project to conduct laboratory research investigations on the physical and chemical properties of medium and fine wools of domestic and foreign origin, and to conduct processing trials of these wools through the principal stages of worsted manufacture. The purposes of this study were to examine the chemical and physical differences between medium and fine wool types, to examine their processing characteristics through worsted manufacture, to study the aging process in detail by research techniques on single fibers and fiber assemblies, and to determine, if possible, whether the beneficial effects of resting the wool during manufacture may be obtained by other means than those requiring long periods of storage at intermediate stages in processing.

Carefully supervised mill trials have permitted a detailed study to be made of the characteristics of an Australian and a domestic fine wool, and a New Zealand and a domestic medium wool, through the processes of scouring, carding, combing, drawing, spinning, weaving, dyeing, and finishing. The principal features of these mill trials, presented in section **II** of this paper, illustrate the behavior of each of these four wools throughout worsted processing into a standard 13-oz. serge fabric. A summary of the research studies at Princeton on the chemical and physical properties of single fibers and the mechanical properties of such fiber assemblies as top, roving, and yarn is presented in section **III** of this paper. Although this work is admittedly not complete, the conclusions which can be drawn at present are summarized in section **IV**.

II—Mill Processing Trials

A—Selection of the Four Wool Types

In the initial stages of planning for the Wool Research Project, the first problem was that of select-

* This article is essentially a progress report of work conducted on the Wool Research Project sponsored by The Wool Bureau, Inc., acting for the International Wool Secretariat and the American Wool Council; by the National Wool Trade Association; by the United States Department of Agriculture (RMA Contract A-ls-30934 supervised by the Western Regional Research Laboratory of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration); by the Office of Naval Research (Contracts Nonr-09000 and Nonr-09001); and by a number of American wool manufacturers.

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ing the proper wools for study. A 64's grade of wool was selected because this grade represents the bulk of the fine wools used and corresponds to the grade specified for use in most Army and Navy fabrics. A 58's grade, or ½-blood wool, was selected because this grade represents the bulk of the medium wools in use and may be considered as a possible minimum standard for military apparel fabrics.

To make each trial lot sufficiently large for proper mill processing, the minimum amount was set at a weight of 1,500 lbs. clean wool. In order to keep these tests at a manageable level, the wool lots were limited to four types: two fine wools of a 64's grade, one of domestic and the other of Australian origin; and two medium wools of a 58's grade, one of domestic and the other of New Zealand origin.

The fine Australian wool was of the merino type grown in Queensland; the domestic fine wool was of the French merino breed, Rambouillet, and was grown at the U. S. Sheep Experiment Station in Dubois, Idaho. The medium New Zealand wool was of the Corriedale type grown in the mid-Canterbury district of New Zealand; matched to this wool was a domestic medium wool of the Targhee type grown also at the U. S. Sheep Experiment Station in Dubois, Idaho. For the two domestic wools, rather extensive background information is available about the breeding stock as well as the geographical location and climatic conditions under which the wool was grown. Information concerning the conditions of growth of the two foreign wools is not readily available. It is known, however, that the fine Australian wool was obtained from two different sheep breeding stations and that the New Zealand medium wool represents a composite sample selected from lots grown by 190 different wool growers. The four animal types are illustrated in Figure 1.

When the various lots arrived at the mill, core samples were taken to establish the yield as well as the grade of each wool type. The average yield of these wools varied from 50% for the fine domestic

FIG. 1. *Sheep breeds of master wools.*

TABLE I. FINENESS MEASUREMENTS ON FOUR MASTER WOOLS

Wool type	Fiber diameter (μ)	U. S. grade
Fine Australian	19.8	70's
Fine domestic	21.1	64's
Medium domestic	26.2	58's
Medium New Zealand	26.3	58's

wool to 58% for the fine Australian wool. All four wools were then sorted, and samples were withdrawn for staple length determination.

In buying the wools, the two main properties specified were fineness and length. A perfect match was sought with respect to these two properties for the two fine wools as well as for the two medium wools. The core samples indicated a difference of a full grade between the fine domestic and the fine Australian wools. Microscope measurements on the four wools, using the short-fiber method, are summarized in Table I.

The fineness of each lot as established by visual sorting, however, did not show up the difference of a full grade between the two fine wools as indicated by the microscope method. According to the visual sorting procedure, the Australian wool was only about 15% finer, or 0.3 μ finer, than the fine domestic wool. The two medium wools, however, were found to be well matched by both the visual inspection and the microscope methods. Photographs of the cross sections of fibers representing each of the four wools (Figure 2) illustrate clearly the range of fineness encountered among the four types.

B—General Outline of Processing Conditions

Prior to the start of the actual manufacturing, it was agreed that each lot should be run through the mill in consecutive order, starting with the Australian wool, followed by the fine domestic, the medium domestic, and the medium New Zealand. The reasons for this were: (1) the Australian wool represented the largest lot and was to be used as standard; and (2) all of the machinery in the plant was geared to operate with optimum efficiency for that type of wool.

It was also agreed that all four wools be processed on the same equipment—the same scouring train, same cards, same gills, same combs, same drawing, spinning (French system), and twisting frames, woven on the same looms, scoured in the same dolly washer, dyed in the same dye baths, and shorn and steamed on the same finishing equipment. The machinery was only to be adjusted for speed of operation when difficulties were such as to impair the production of a proper, uniform yarn.

A 13-oz. serge (standard 2/2 twill, 70 ends and picks per in.) was selected as the fabric to be woven for study. The weaving specifications included the following: 8 harnesses, 60 picks per in., 70-yd. pieces, 4,050 ends, reed width 71 in., with 40 ends in the selvage reversed drawn in. This construction made it necessary to spin 1/40 worsted yarn. It was realized that there would be no trouble with spinning of the two fine wools, but that some difficulty might arise in spinning a 1/40 yarn with the medium wools because this yarn count was considered to be very close to the possible spinning limit for these wools.

The main sampling stations during the manufacturing processes were selected to include *top, roving, yarn, grey fabric, ready-to-dye fabric,* and *finished fabric.* These stations followed the combing, drawing, spinning, weaving, wet finishing, and final finishing operations, respectively.

C—Scouring

The scouring process revealed the first significant differences in behavior among the four wools. The

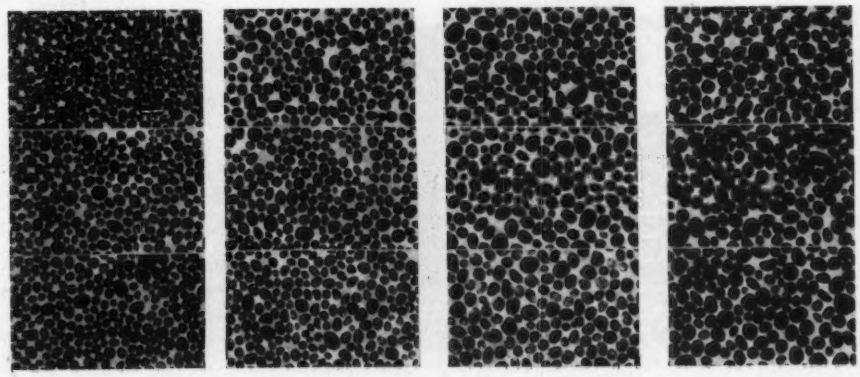

FIG. 2. *Photographs of cross sections of fibers representing the four wools. From left to right: fine Australian, fine domestic, medium domestic, and medium New Zealand wool.*

two fine wools were scoured one day, and the two medium wools on the following day. In both instances the operation was started with fresh solution, using exactly the same amount of soap and soda.

The efficiency of the scouring is revealed by the amount of extractable matter left in the wool after the scouring process. The mill man considers a clean wool one which has about 0.75% residual ether-extractable material. Wool which has a residual amount of ether-extractable material above 1% is considered to be poorly scoured, while wool which has less than 0.5% ether-extractable material is assumed to be overscoured. The results shown in Table II and Figure 3 indicate that (*a*) the two domestic wools have average scouring characteristics, (*b*) the Australian wool is overscoured, and (*c*) the medium New Zealand wool is poorly scoured. In order to decrease the average residual grease content on the New Zealand wool, additional soap and soda were introduced into the scouring bath toward the end of the scouring operation.

D—Carding, Combing, and Drawing

Before undertaking the carding operation, a decision was required concerning whether the wools should be processed on the new metallic worsted cards, clothed with metallic wire throughout, or on the standard double-cylinder worsted cards with wire clothing. The Research Advisory Committee decided in favor of using the new metallic clothing cards.

A further delay in the processing was required in order to give the Textile Research Institute staff enough time to make a preliminary study of the aging process and to suggest an aging time for the master wools in manufacture. After several months of preliminary work, the staff recommended that approximately 3 weeks appeared to be the optimum resting time and suggested this as the aging period.

After all this preliminary work was completed, the manufacturing operation was started. In the combing of the wools, the main concern was with regard to the change in the average fiber diameter and the role which fiber length plays. The carding and combing operations followed the standard procedure of the French system: carding, three gillings, combing, assembling, backwashing, and final gilling. No changes whatsoever had to be made from the original setting used for the fine Australian wool.

The fineness measurements made by the microscope method on the core samples, card slivers, and tops revealed an excellent correlation in these properties among the results obtained in the various stages. Figure 4 and Table III clearly indicate this correlation.

TABLE II. RESIDUAL ETHER- AND ALCOHOL-EXTRACTABLE MATERIAL ON FOUR MASTER WOOLS AFTER SCOURING

Wool type	% Ether-extractable material	% Alcohol-extractable material
Fine Australian	0.27	0.88
Fine domestic	0.67	0.89
Medium domestic	0.69	0.99
Medium New Zealand	1.38	0.94

TABLE III. FINENESS MEASUREMENTS ON THE FOUR MASTER WOOLS IN RAW WOOL, CARD SLIVER, AND TOP STAGES

Wool type	Fiber fineness (μ)		
	Raw wool	Card sliver	Top
Fine Australian	19.8	19.5	19.8
Fine domestic	21.1	21.3	21.8
Medium domestic	26.2	25.6	26.5
Medium New Zealand	26.3	26.4	26.2

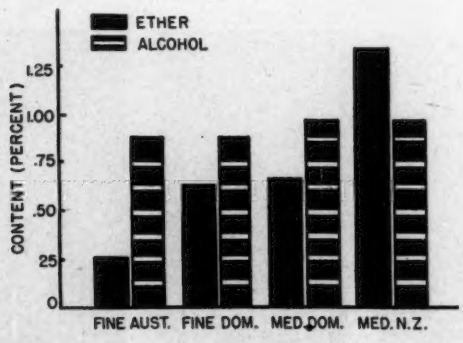

FIG. 3. *Analysis of scoured master wools; extractable matter.*

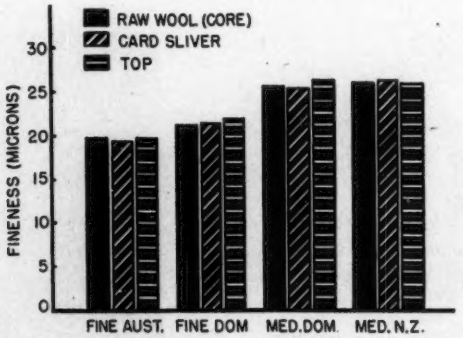

FIG. 4. *Fiber fineness through processing of master wools.*

TABLE IV. STAPLE LENGTH MEASUREMENTS ON THE FOUR MASTER WOOLS IN RAW WOOL, CARD SLIVER, AND TOP STAGES

Wool type	Staple length (in.) Raw wool	Card sliver	Top
Fine Australian	2.6	1.8	2.4
Fine domestic	3.0	2.2	2.8
Medium domestic	3.3	2.8	3.2
Medium New Zealand	3.4	2.9	3.3

The results show that the core-boring method of sampling is not only an excellent tool for determining the shrinkage of raw wool, but it also has proved to be a good method for obtaining samples to establish the fineness or grade of the wool. The card sliver is in three instances finer than the top; this result is reasonable inasmuch as a certain percentage of finer, shorter fibers are removed in the combing process.

The important role which staple length plays in worsted combing is clearly seen from the data in Table IV and Figure 5. One of the questions to which a definite answer was being sought was, "What length must the raw wool be in order to produce a specified top length?" For example, the comber wants to produce a 3-in. top; what, then, should the length of the raw wool be when selected by the wool buyer? The answer is that the average staple length must be at least equal to the specified top length.

In order to establish the foregoing results, it was first necessary to develop an acceptable test procedure for determining the staple length of a wool lot. Indeed, a standard procedure for establishing the top length has been developed, but it was recognized that the commercially used method of determining the raw staple length by grasping the staple in both hands and stretching it is not precise, due in part to its subjective nature. Mr. Elroy Pohle* of the Wool Section of the United States Department of Agriculture has demonstrated that staple length may be determined acceptably by measuring the length of the unstretched raw wool staple.

The average length of all four wools in the form of card sliver is shorter by approximately ½ in. than the final top length. This reduction in length is caused by fiber breakage in scouring and in carding; the increase in the measured length of the wool in the top stage is due to the combing operation, in which the short and broken fibers are selectively removed. By reference to the data in Table V, it may be seen that the quantity of short fibers removed increases with the grade of the wool.

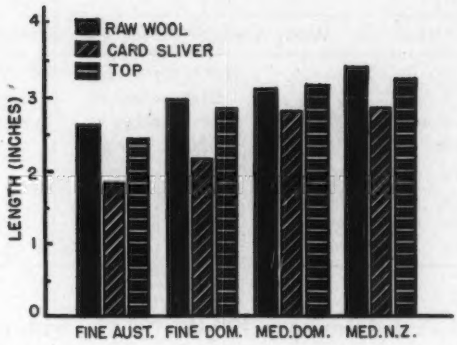

FIG. 5. *Fiber length through processing of master wools.*

The term "tear" is normally used in England and on the continent, while the average American mill man expresses the noilage as a percent of the wool processed. The noilage figures for the four wools according to this method of evaluation are: fine Australian wool, 9%; fine domestic wool, 8%; and 6% for both of the medium wools.

Following the production of top, it was decided that the wool should have a resting period of about 1 week in order to give the top an opportunity to come to moisture equilibrium. During carding and combing, the average room tempertaure was maintained at 80°F and the average relative humidity at 70%. The temperature in the top cellar was maintained at 70°F and the relative humidity at 70%.

In the combing and drawing departments, no adjustments were made in the machinery settings; the original settings used for the fine Australian wool were maintained. The roving was drawn to a

TABLE V. LENGTH AND TEAR DATA FOR FOUR MASTER WOOLS

Wool type	Length (in.)	Tear (ratio of top to noils produced)
Fine Australian	2.4	10.4
Fine domestic	2.8	12.0
Medium domestic	3.2	15.6
Medium New Zealand	3.3	15.5

* Johnston, D. D., Ray, H. D., Manning, W. J., and Pohle, E. M., "Relationship of Staple Length in Grease Wool and Resultant Top," Production and Marketing Administration, U. S. Dept. of Agriculture, June 1951; TEXTILE RESEARCH JOURNAL **21**, 757-62 (1951).

TABLE VI. WOOL GRADE *Versus* SPINNABILITY

U. S. wool grade	Ave. fineness (μ)	Max. spinning Number Worsted	Yds./lb.	No. of fibers in yarn cross section
70's	20.3	70	39,200	30
64's	21.8	60	33,600	30
60's	24.0	50	28,000	30
58's	26.3	46	25,760	27

weight of 4 drams per 40 yds., which, with a draft of 8, reduced this roving to a 40's worsted yarn.

E—Spinning and Weaving

As a prologue to the analysis of the spinning trials on the four master wools, it will be instructive to consider the relationship between wool grade and spinnability in the form of data (Table VI) originally compiled by Kennedy.*

To illustrate the applicability of this material to the present study, data for only four grades of wool are shown here. In his original tabulation, Kennedy showed that the maximum spinning number on the Bradford system for a 70's grade wool should be a 70 worsted; for a 64's grade wool, a 60 worsted; for a 60's grade wool, a 50 worsted; and for a 58's grade wool, a 46 worsted. For each of the four types of wool considered in Table VI, it can be shown easily that, at the maximum count, there are 30 fibers in the yarn cross section. Not all spinners, however, agree that this is the correct number. Most European spinners have found that the economical maximum spinning limit is reached with a minimum of 40 fibers in the yarn cross section.

The change in spinnability in the range from an 80's grade wool to a 50's grade wool is illustrated in Figure 6. Two curves relating worsted count to average fineness are shown, one for 30 fibers and the other for 40 fibers in the yarn cross section. By reference to Figure 6, it is clear that an 80's grade wool with 30 fibers per yarn cross section will spin to an 80 worsted, while the same wool grade with 40 fibers per yarn cross section will reach maximum spinning limit at 60 worsted, a count which is 20 numbers coarser.

In the present study, the wool lots mentioned above included a 70's, a 64's, and two 58's grades. It may be seen from Figure 6 that no difficulty should be

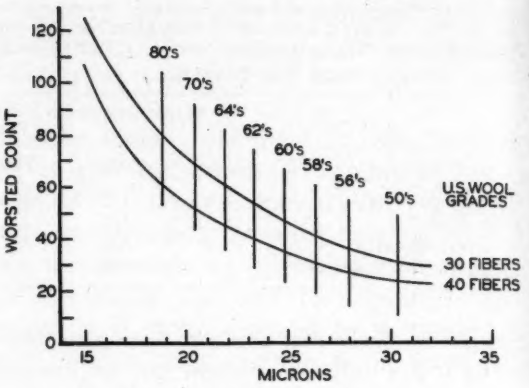

FIG. 6. *Wool grade* vs. *spinnability.*

encountered in spinning a 1/40 worsted with a 70's grade wool and 40 fibers per cross section, since that combination should be capable of spinning up to a 1/52 yarn. Also, a 64's grade of wool with 40 fibers per cross section should easily provide a 1/40 yarn; a 58's grade with 30 fibers per cross section should spin to a 1/42 yarn, and with 40 fibers per cross section the spinning limit should drop to a 1/30 yarn.

Figure 7 shows the average number of fibers found when 20 single yarns of each of the four master wools were analyzed by using the cross-section method. By actual measurement of these four cross sections, the number of fibers for the fine Australian wool was found to be 53; for the fine domestic, 41; medium domestic, 29; and medium New Zealand, 27. By reference to Figure 6, it is found that the predicted number of fibers should be 30 per cross section of the yarn for a 58's grade of wool spun to 1/40 yarn, and the predicted number should be 55 fibers for the fine Australian wool (70's), and 44 for the fine domestic wool (64's). In each instance, the actual number of fibers per cross section was below the predicted figure for the grade of the wool.

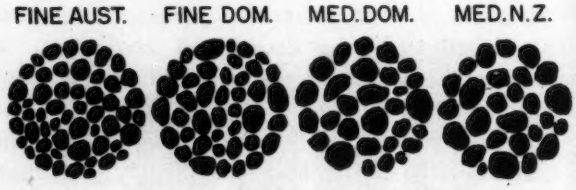

FIG. 7. *Cross sections of 1/40 (Grex 221) worsted yarns made from master wools. Fiber fineness and number of fibers observed in cross sections: fine Australian wool, 19.8 μ, 53 fibers; fine domestic wool, 21.6 μ, 41 fibers; medium domestic wool, 26.1 μ, 29 fibers; medium New Zealand wool, 26.1 μ, 27 fibers.*

* von Bergen, W., and Mauersberger, H. R., "American Wool Handbook," 2nd edition, New York, Textile Book Publishers, Inc., 1948, p. 586.

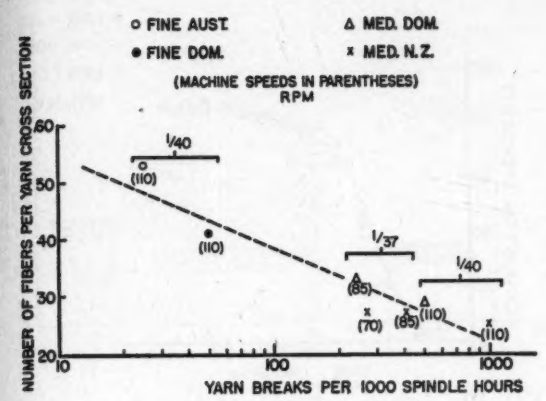

FIG. 8. *Spinning performance of master wools; worsted yarn number.*

With the SACM * spinning frame set to operate at a front-roll speed of 110 r.p.m., the number of ends down per 1,000 spindle hours was found to be 25 for the fine Australian wool and 50 for the fine domestic wool. In the case of medium domestic wool, however, the number of ends down on the spinning frame was ten times that for the fine domestic wool, or 500 breaks per 1,000 spindle hours. The spinning performance of the New Zealand wool was even worse than that of the medium domestic wool, running at over 1,000 breaks per 1,000 spindle hours. In view of this poor spinning behavior for the medium wools, it was agreed that the yarn count should be reduced to 1/37. This reduction in yarn count, together with a decrease in machine speed from 110 to 85 r.p.m., resulted in reducing the number of ends down on the spinning frame from 500 to 240 breaks per 1,000 spindle hours for the medium domestic wool, and from 1000 to 410 breaks per 1,000 spindle hours for the New Zealand medium wool. In the case of the New Zealand medium wool, the machine speed was further decreased from 85 to 70 r.p.m., with an attendant drop in ends down from 410 to 270 per 1,000 spindle hours. Even by spinning the medium wools to a 1/37 yarn, the percentage of breaks was found to be far greater than 10%, or 100 breaks per 1,000 spindle hours, considered acceptable by many spinners. By reference to Figure 8, it can be seen that reducing the yarn count from 1/40 to 1/37 worsted increased the average number of fibers per cross section from 29 to 33 for the medium domestic wool and from 25 to 27 for the medium New Zealand wool.

* Société Alsacienne de Constructions Mécaniques.

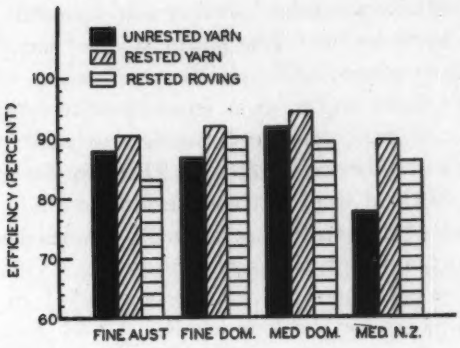

FIG. 9. *Weaving data of master wools; loom efficiency.*

The spinning performance of the rested roving for the medium domestic wool improved considerably over that of the fresh roving; the number of ends down on the frame decreased from 240 to about 170 breaks per 1,000 spindle hours. By resting the roving made from the New Zealand wool, the number of ends drown dropped only from 270 to 250 per 1,000 spindle hours.

The performance of the four wools in weaving is illustrated by the bar graph in Figure 9. These results show very clearly that the rested yarn in all four instances had the highest weaving efficiency; in every case the weaving efficiency was 90% or higher. There is no clear indication of the influence which resting the wool in the roving may have on its weaving performance. It is seen in two instances that the yarn from the rested roving showed better weaving performance than unrested yarn. Although in most instances the yarn from the rested roving also had resting periods in the yarn stage, amounting in some cases to as much as 30 days, differences in weaving performance are still apparent between rested roving and rested yarn. The results indicate that storing the yarn in the yarn cellar for 3 weeks prior to weaving results in a 2% increase in loom efficiency.

F—Dyeing and Finishing

In the finishing of the cloth no special obstacles were encountered. In order to produce the proper fabric weight, a correction had to be made in the original layout simply because this layout included a short fulling operation. Since fulling would introduce an additional problem for study, it was decided to by-pass this operation and simply wash, crab, dye, shear, and finally decate the fabric. The fabrics were

washed with a regular low-titer soap in a dolly washer with wash loads of four pieces at one time.

Ninety-seven fabric pieces were woven. Thirty-three pieces were made from the fine Australian wool, 20 from the fine domestic wool, 23 from the medium domestic wool, and 21 from the medium New Zealand wool. It was decided to study the influence of the resting periods on the finished cloth and process four pieces of each group. This meant that for each wool 3 × 4 pieces were used, or a total of 48 pieces for the four wools. All 48 pieces were dyed with the same specially prepared metallized dye made and contributed to this work by General Dyestuff Corporation.

The total time of about 4 hrs. was required for the dyeing operation: approximately ¾ hr. for wetting out and adding the dye and acid, 1½ hrs. to reach the boil, and 2 hrs. at the boil. The dyeing was started at a temperature of 130°F. Approximately 1 g. of sulfuric acid per liter (8% by weight) was used with the dye; no leveling agent was added.

By reference to Figure 10, it is seen that the exhaustion rates were comparable for all four wools, reaching an average of 77% exhaustion at the boil and a maximum exhaustion of 85% after 2 hrs. at the boiling temperature. An amazing difference in color depth, in one way expected, was observed on comparing the four fabrics. By careful measurement it was found that the fine domestic wool had a depth of shade after dyeing about 5% darker than the fine Australian wool, and the medium wool pieces had a depth of shade about 20% darker than the fine Australian wool. When four pieces, one of each type, were dyed together in the same dye bath, these color differences were magnified so that actually the maximum difference between the fine Australian and the medium wool was about 30%. This illustrated clearly that the origin of some of the troubles in piece dyeing may stem from an unfortunate interchange of yarns of the same count but of different wool grades. The differential shading of the various wool grades results in contrasting bars in the completed fabric.

The same marked difference in shade was produced with fine Australian and medium domestic wools dyed a navy color in the same dye bath. The reason for this difference in shade has been attributed, up to now, to the difference in specific surface of two wool grades. The total surface to be dyed in the fine Australian wool (70's) is 30% greater

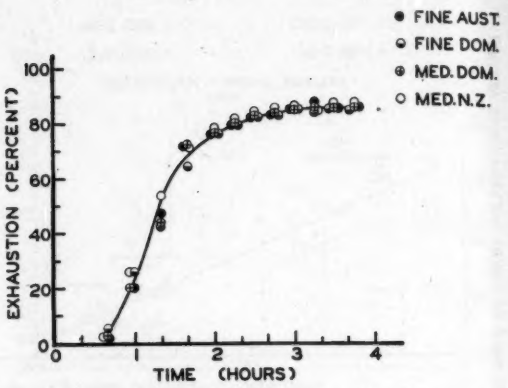

FIG. 10. *Dyeing exhaustion rate of master wools.*

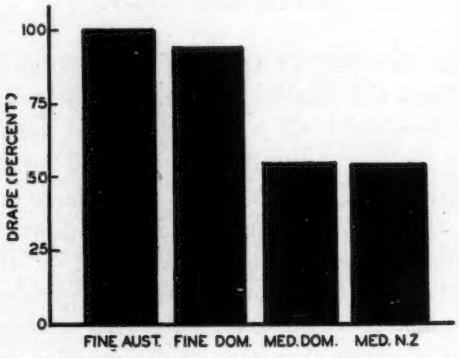

FIG. 11. *Drape of finished pieces.*

than that for either medium wool. It is argued, therefore, that it should be 30% lighter in shade. Since the finer wools have more surface to reflect the light, the sensation of a lighter shade is produced. Reflection measurements have proved that this is true. It is also argued that for coarser wools, it takes a longer time to penetrate with dyes; therefore, the interior of the fiber may not be quite as dark as the outer ring. Fiber-section studies indicate that this is not the case, as the fibers are all dyed uniformly from surface to center. It would appear, however, that further explanations for this shade difference are necessary before an unequivocal answer to this problem may be given.

G—Drape Characteristics of Fabrics Made from Four Wools

Figure 11 illustrates the draping characteristics of fabric pieces made from the four wools. Drape is related closely to the stiffness of the fabric; drape and the ability to fall into folds are synonymous. It is not difficult for the fabric experts as well as for most amateurs to feel the small difference which

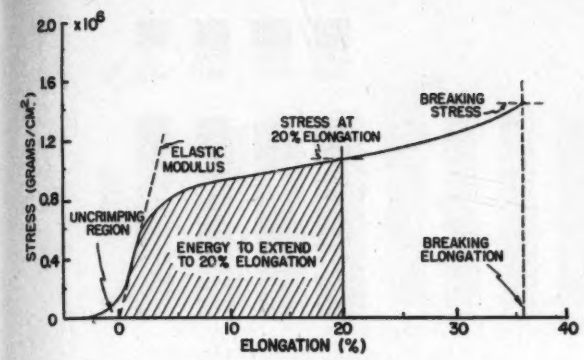

FIG. 12. *Typical stress-strain curve for single wool fiber.*

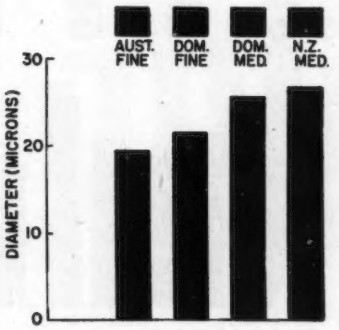

FIG. 13. *Single-fiber properties; average diameter (top, roving, yarn).*

exists between the fine Australian and the fine domestic wool. No discernible difference was found between the fabrics made from the two medium wools. Everyone agrees, however, that a very large difference exists between the fine and medium wools.

Fabrics from the four master wools were tested by the Fabric Research Laboratories using the Drapemeter technique developed by Chu, Cummings, and Teixeira.* This drape test was conducted by draping the fabric over a circular horizontal disk, and then measuring the area within the projected contour of the draped fabric. The results are illustrated in Figure 11, in which the differences in draping characteristics are shown in bar graph form, adjusting the actual values to make the fine Australian wool 100%. Those who have seen the Forstmann-Textile Research Institute exhibit † of the four wool types will recognize that the fine Australian wool falls from its support more abruptly and in more folds, thereby covering a smaller projected area, than the fine domestic wool or, in even greater contrast, than either of the medium wools. The increase in projected area characterizes a lower draping ability, and the drape index on the ordinate of Figure 11 is inversely proportional to that projected area.

III—Research Studies on Master Wools *

The research work at Textile Research Institute on the physical and chemical properties of each of four master wools and on the studies of their behavior throughout processing and throughout the aging period divides itself logically into four distinctly separate categories. First, the basic physical properties, such as elastic modulus, breaking stress, and breaking elongation, indicate that there is a strong similarity in these properties among the four wool lots. Second, these four wools, insofar as their physical and chemical properties are concerned, behave similarly throughout the stages of worsted manufacture. Third, the mechanism of the aging process, as studied by measuring the physical properties of such fiber assemblies as top and roving, is shown to be related to the relaxation of stresses imposed upon the fibers by previous processing operations. Fourth, the aging process, currently carried out under room temperature conditions of 70% R.H. and 70°F, may be accelerated by a steaming treatment both in the top and in the roving stages.

A—Comparison of Physical and Chemical Properties of Four Master Wool Lots

As a basis for comparing the physical properties of the four master wools in tensional strain, stress-strain curves were obtained at a constant rate of strain for large numbers of single fibers withdrawn from each of the wool lots at the following representative stations throughout processing: grease wool, scoured wool, top, roving, yarn, and grey, ready-to-dye, and finished fabric. A schematic drawing of a typical stress-strain curve for a single wool fiber is shown in Figure 12. All of the fibers were tested at 65% R.H. and 70°F; 1-in. samples were used and extended at the rate of 0.5 in. per min. The principal properties considered in comparing the physical properties of the four wool lots are the following:

* Chu, C. C., Cummings, C. L., and Teixeira, N. A., TEXTILE RESEARCH JOURNAL **20**, 539–48 (1950).

† Displayed at the twenty-second Annual Meeting of Textile Research Institute held at the Hotel Statler in New York City, November 8 and 9, 1951.

* A more complete summary account of the research studies on the physical and chemical properties of the four wools will be published soon. This will include descriptions of the various new techniques developed for these studies.

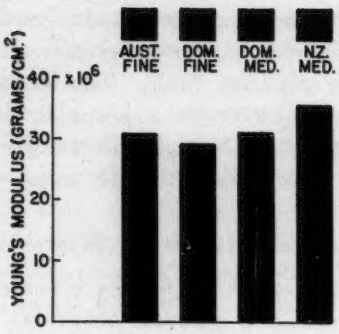

FIG. 14. *Single-fiber properties; Young's modulus (top).*

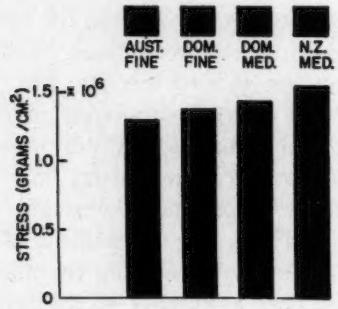

FIG. 15. *Single-fiber properties; stress at break (top).*

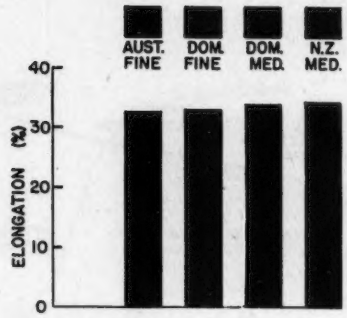

FIG. 16. *Single-fiber properties; elongation to break (top).*

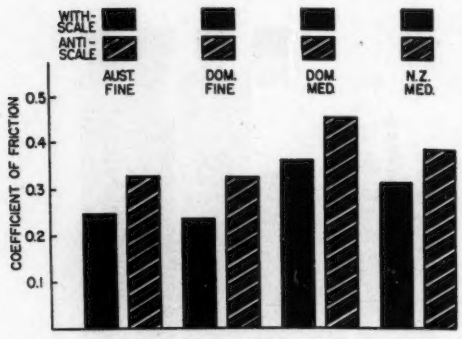

FIG. 17. *With-scale and anti-scale frictional properties of master wools.*

fiber diameter, elastic (or Young's) modulus, stress at 20% elongation, energy to extend the fiber to 20% elongation, breaking stress, and breaking elongation. It develops, however, after a lengthy comparison of physical properties, that the largest difference among the four lots of wools appears to be in their mean fiber diameters. Figure 13 shows the mean diameter values for single fibers obtained from the four lots of wool and averaged over the top, roving, and yarn stations in processing. The largest difference in diameter, about 40%, occurs between the Australian fine wool and the New Zealand medium wool. The physical properties which we shall refer to for a comparison of the four wools are those in which the differences in cross-sectional area are removed by dividing each measured force-dependent property by the cross-sectional area of each fiber and reducing the properties to force per unit cross-sectional area. In the case of Young's modulus (Figure 14) and the stress at break (Figure 15), both properties referring to a unit cross-sectional area, there was found to be no difference among the four master wools as great as that existing among their diameters. Note that the elongation to break (Figure 16) has almost the same value among the four wools in the top stage. Of course, this is a property which requires no reduction to unit cross-sectional area. The other physical properties shown in Figure 12 exhibit similar behavior.

Having found that the basic physical properties on a unit cross-sectional area basis are comparable among the four wools, some confidence is gained in the conclusion that the wool lots, although grown in widely different sections of the world, behave in a strikingly similar manner when account is taken of their differences in fiber size or diameter. This conclusion, based on a thorough study of the tensional properties of fibers withdrawn from each of the four master wool lots, is fortified by the results of chemical studies on these wools. Insofar as cystine and amino acid content as well as alkali solubility are concerned, the wools are also strikingly similar.

One of the physical properties, however, which should influence processing importantly and should depend on the surface properties of fibers withdrawn from each of the master wools is the coefficient of

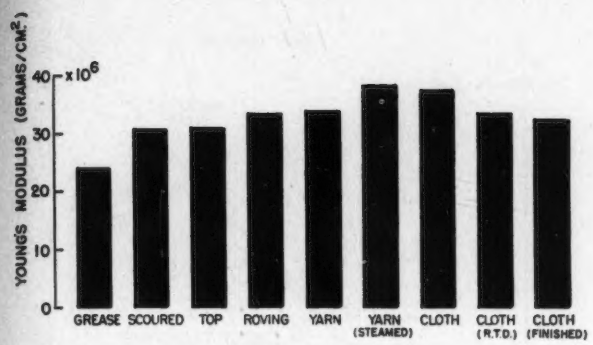

FIG. 18. *Single-fiber properties; Young's modulus (progression through processing—domestic medium wool).*

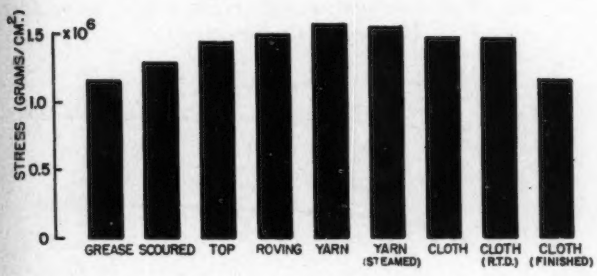

FIG. 19. *Single-fiber properties; stress at break (progression through processing—domestic medium wool).*

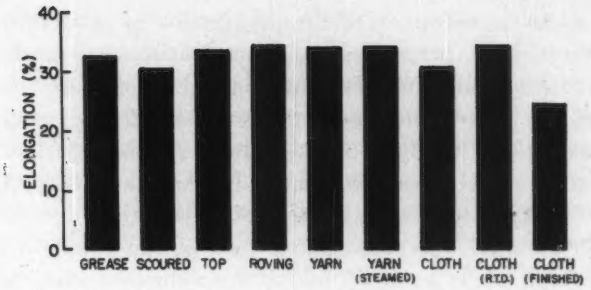

FIG. 20. *Single-fiber properties; elongation to break (progression through processing—domestic medium wool).*

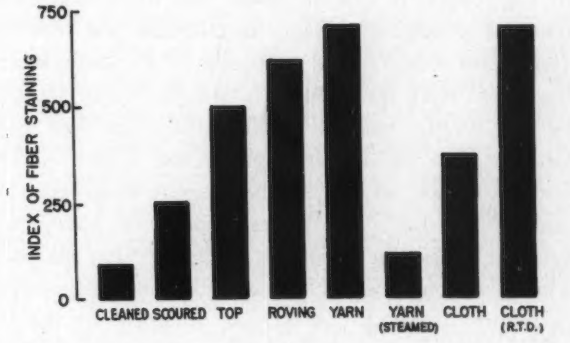

FIG. 21. *Wool fiber staining by Pauly reagent (progression through processing—domestic medium wool).*

friction. The frictional properties of fibers withdrawn from the four wools were measured against a wool felt in a manner similar to the technique used by Harris and coworkers.* Measurements of the with-scale and anti-scale frictional properties of these wools (Figure 17) show that, in general, the fine wools have lower with- and anti-scale frictional coefficients than the medium wools, the difference between the with- and anti-scale direction being roughly comparable for all the master wools. It should be noted that the domestic medium wool has the largest with- and anti-scale frictional coefficients and that there is no obvious relationship between the mean diameter of the wool lots and their coefficients of friction.

B—Comparison of Physical Properties of a Single Wool Lot of Domestic Medium Wool Through Worsted Processing

Since the principal physical properties were found to be similar among the four wool lots, it was considered important to find out how these properties changed throughout the worsted processing operations from the grease wool to the finished cloth. The same tensional properties were followed throughout processing as were used to compare the four wools at the top stage: Young's modulus (or a measure of the low-elongation stiffness), the stress at 20% elongation, the energy to extend to 20% elongation, and the limit properties—stress at break and elongation to break. In general, each of those derived or reduced properties appears to increase through physical processing up through the yarn stage and to decrease from grey through ready-to-dye to finished cloth. The changes in Young's modulus, stress at break, and elongation to break for domestic medium wool are illustrated in Figures 18, 19, and 20, respectively. It is important to note that each of the four wools examined shows the same type of progression through processing. The domestic medium wool lot was chosen for discussion here because the data are more complete on this wool than on fibers from other wool lots.

*Frishman, D., Smith, A. L., and Harris, M., TEXTILE RESEARCH JOURNAL **18**, 475–80 (1948).

During the study of the progression of properties throughout processing, an examination was made of the surface modification imposed on each of the wools by both the physical and chemical processing conditions on the worsted system. The degree to which a fiber could be stained by the Pauly reagent was used to estimate surface modification due to processing. On this index, each of the wools appeared to be modified in the same manner starting from the laboratory cleaned stage (alcohol-ether extraction), through the yarn stage, and finally to the grey and ready-to-dye fabric stages. By reference to Figure 21, it will be noted that there is a rapid increase through the stages of cleaning and scouring (soap and soda) to the unsteamed yarn stage, where the fibers have the greatest susceptibility to staining by the Pauly reagent. After the yarn has been steamed (to set in the twist), the ability of the fibers to take up the Pauly reagent is greatly reduced. In fact, the fibers appear to have been brought back to a condition similar to that for wool which has only been cleaned with the alcohol-ether extraction method. A further increase in surface modification, however, is evident throughout fabric manufacture, particularly in the grey and ready-to-dye states. Observations on the scale condition indicate that as the fibers are brought through processing up to the yarn stage, the scales on the surface of the fiber become progressively upturned. The steaming process lays these scales down again, with the attendant effect of decreasing the ability of the fiber to accept a surface stain. This observation led to the conjecture that the rate of dyeing of these wools might also be affected by the steaming process in the yarn stage and might also reflect differences in surface modification throughout processing as revealed by the Pauly test. Laboratory bulk dyeing tests showed that the rate of dye uptake, for both an acid and a neutral dye, is proportional to the index of fiber staining by the Pauly reagent. The progression of rate of uptake as a function of stage in processing is not new with this laboratory and has been reported at some length by Royer and coworkers.* However, the striking effects of steaming on both the uptake of Pauly reagent on the surface of the fiber and the uptake of dye in bulk tests are, it is considered, novel observations.

* Royer, G. L., Millson, H. E., and Amick, C. A., *J. Soc. Dyers Colourists* **63**, 214–24 (1947).

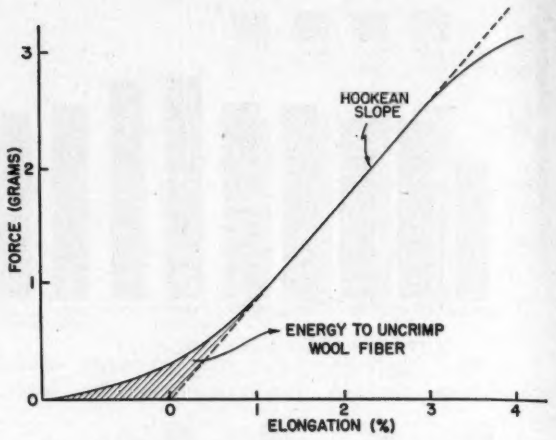

FIG. 22. *Force-elongation curve for single wool fiber (low-elongation region).*

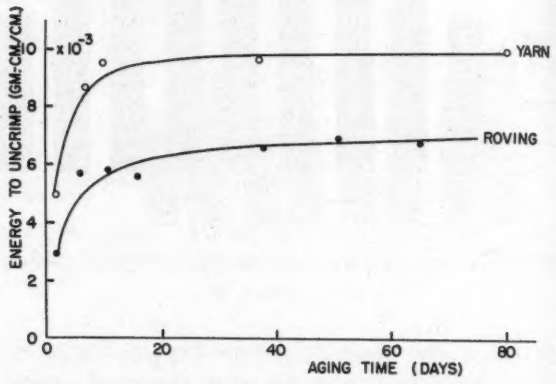

FIG. 23. *Effect of aging on energy to uncrimp single wool fibers (New Zealand medium wool).*

C—Studies of the Aging Process

Each one of the physical properties, such as Young's modulus, stress at 20% elongation, energy to extend to 20% elongation, stress at break, and elongation at break, was followed on all of the four wool lots throughout the aging period after the manufacture of top, roving, and yarn. No significant differences in these properties throughout the resting period were observed. This negative result suggested rather strongly that the aging effect might be connected with the cooperative nature of the behavior of fibers in fiber assemblies and that some clue to this situation might be obtained by studying the crimp characteristics of these wools through a time cycle similar to that of aging in industrial manufacture. In order to conduct these measurements on single fibers, the very low elongation region, depicted schematically in Figure 22, was examined, and the

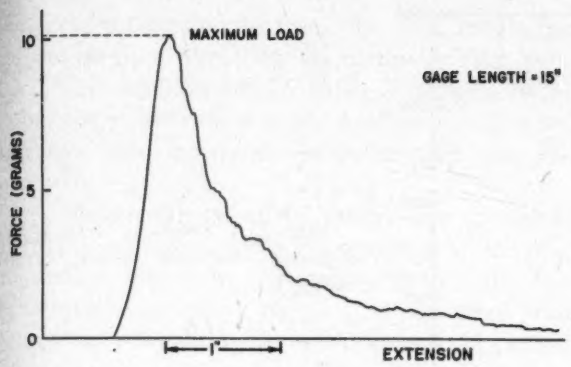

FIG. 24. *Typical force-extension curve for roving (domestic medium wool).*

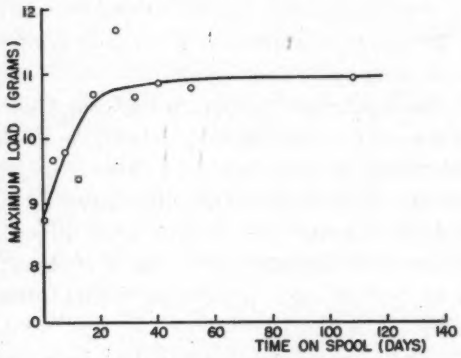

FIG. 25. *Change in maximum load with time of aging on spool (domestic medium wool, roving).*

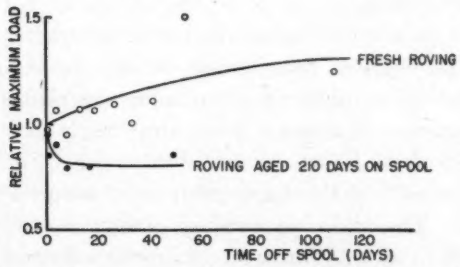

FIG. 26. *Change in maximum load with time of aging after unwinding from spool (domestic medium wool, roving).*

energy to uncrimp (shaded area) single fibers was measured progressively after the manufacture of top, roving, and yarn. Figure 23 shows results of these measurements on representative single fibers of New Zealand medium wool, which indicate the effect of aging on the energy to uncrimp the fibers as a function of time after manufacture. The energy to uncrimp single fibers appears to be the only single-fiber property measured to date which reflects changes occurring in the aging process as conducted in industrial manufacture.

Measurements of the physical properties and behavior of top and roving suggested themselves as fruitful areas in which to study effects of the aging process. Observations on fresh and aged top indicated that the former tends to expand laterally (or spread) if it is unwound from a ball and left to stand unconstrained on a smooth surface. The latter or aged top has little, if any, tendency to spread if unwound after an aging period of several months on the ball at 70% R.H. and 70°F. Physical measurements of the controlled drawing of roving itself, illustrated schematically in Figure 24, have pointed to the importance of the time variation of changes in maximum load to which roving may be extended as well as the low-elongation stiffness of the assembly. Top and roving were found to behave similarly in their aging characteristics and, as an example of this behavior, a typical curve for the change in maximum load for roving aged on the spool up to 120 days is shown in Figure 25. The behavior of the maximum load for roving aged off the spool is shown in Figure 26, where the behavior of fresh roving and aged roving is compared. Note that the fresh roving shows a gradual increase in maximum load with time, while the aged roving shows a decrease in maximum load with time.

D—Pretreatment of Roving with Steam

Since aging appears to be a relaxation of both the constraints due to winding the top on a ball or the roving on a spool and those stresses imposed upon the fibers by previous processing operations, it was felt worth-while to see how an increase in temperature for a short time would affect the spreading behavior of top and roving. Inasmuch as fresh steamed top and roving behaved on spreading similarly to normal top and roving aged at 70% R.H. and 70°F, it was decided further to see whether the steaming condition could produce the same beneficial effects in spinning as normal aging does when carried out at room temperature conditions of 70% R.H. and 70°F. Accordingly, roving from the New Zealand medium wool lot was segregated, and the roving steamed under conditions similar to those for steaming the yarn (30 min. in worsted steaming, 185°F dry bulb, 175°F wet bulb). The first experiments run with fresh roving on the same spinning frame showed that the steaming process decreased

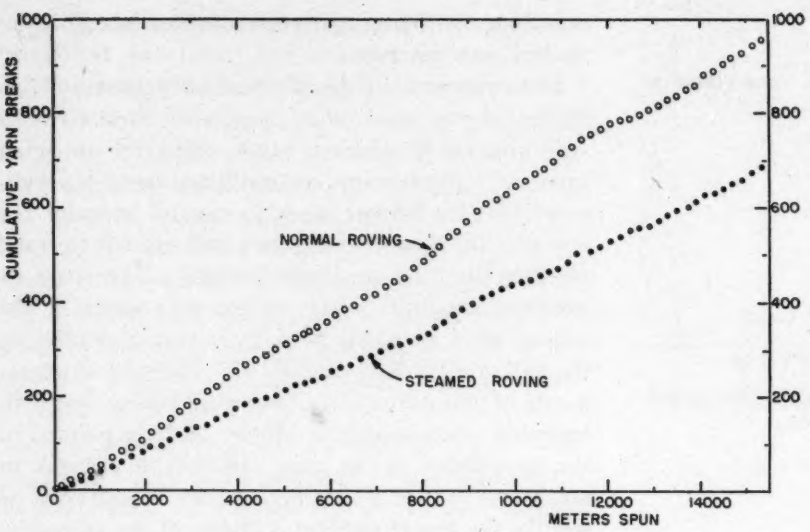

FIG. 27. *Spinning trial (New Zealand medium wool, rested roving).*

the number of ends down by approximately 20%. The same steaming cycle was imposed upon roving which had been rested for 3 weeks, and a spinning trial indicated clearly that steaming the roving under these conditions produced a 30% decrease of ends down on a spinning frame (Figure 27). Certainly the effect of the steaming procedure has not been properly investigated as yet. Preliminary experiments appear to confirm the fact that the steaming process produces the same beneficial effects in processing as aging the wool, and the steaming process can be conducted in a relatively short time with equipment which is presently at hand in the textile industry.

IV—Summary and Conclusions

(1) The aging of wool during worsted processing has been found to be of commercial importance. After aging the roving and yarn at 70°F and 70% R.H. for a period of 3 weeks, an increase was observed in the efficiency of the spinning and weaving operations, respectively. Preliminary studies indicate that this aging period can be shortened by a steaming process.

(2) The average diameter or fineness of a wool lot appears to be the most important characteristic of the wool lot insofar as the efficiency of manufacturing operations is concerned. The average diameter plays a very important role in the drawing, combing, spinning, and dyeing operations. These processing operations depend critically upon the diameter of the fibers in a wool lot regardless of the source of the wool.

(3) In support of the conclusion that fiber diameter in a wool lot plays the dominant role in the level of processing, it was found by laboratory research studies that there are no large differences in the principal physical properties of each wool lot when corrected to unit diameter or in such chemical properties as cystine and amino acid content and alkali solubility.

(4) The core-boring method has been found to provide an excellent means of obtaining samples for the establishment of the fineness of a wool lot prior to processing.

(5) A raw wool which had been scoured to 0.25% free fat content was found to behave excellently insofar as combing and spinning operations were concerned. It appears from this result that reducing the free fat content to below the 0.75% limit does not affect the subsequent processing adversely.

(6) The only processing operation which appeared to depend importantly on the influence of environment or source of the wool is scouring, in which it was found that under identical conditions of soap-soda scouring the Australian fine wool was left with a residual grease content of 0.27%, the two domestic wools (grown in Idaho) with 0.68%, and the New Zealand medium wool with 1.38%.

(7) In the spinning operation, the maximum limit for a French worsted yarn was found to be reached when the yarn contained approximately 40 fibers per cross section and not 30 as generally assumed.

(8) In the studies of dyeing, it was shown that one quality grade difference will result in approximately a 5% difference in shade. A difference of

four grades, existing between the fine Australian and the medium wools studied in this project, produced a shade difference of 20%. Furthermore, in dyeing pieces from the fine and medium wools in the same dye bath, a 30% difference in shade was encountered.

(9) Measurements on the progressive energy to uncrimp wool fibers during the period in which they are resting free from constraints confirm the fact that the gradual return of crimp is connected with the general relaxation effects encountered in normal aging.

(10) Steaming a worsted yarn was found to decrease the rate of dye uptake of the wool as well as the ability of the wool fibers to accept a Pauly stain. Microscope studies of the surface structure of wool fibers show that the steaming process promotes the laying down of scales which have been upturned in previous processing operations. The observed decrease in the rate of uptake of a dye or the ability of the fiber to accept a stain appears to show that the distal edges of the scale and not the surfaces of the scales are regions which control the penetration of dye into the fibers.

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Properties of Fabrics Made from Several Synthetic Fibers and Their Impact on the Textile Industry

G. K. Lake*

IT SEEMS to me that the most important thing that the textile industry has discovered, and which it still has to make clear to all concerned, is the tremendous difference between the fibers that in the past were sometimes referred to as "synthetic fibers," which suffered from the unpleasant connotations of the German word "ersatz," and the new synthetic fibers that we deal with today. It is a matter of great concern to all of us who are interested in the development of new synthetics when we hear such expressions as "synthetic wool" and "artificial silk," or such absurd concepts as "a new type of cotton made from peanuts." The distinction between the new synthetics and the old synthetics becomes much clearer when one gives some consideration to the historical background.

There was, I think, no question in the mind of Chardonnet when he carried out his first experiments in the nineteenth century that he was attempting to produce an artificial silk. I do not believe that any of the founding fathers of the rayon industry had in their minds the goal of producing a new textile. Silk was extraordinarily expensive and was the most desirable fabric available. The very methods that they explored betrayed their thinking. They were attempting only to duplicate mechanically the natural processes of the silkworm. Regardless of the various methods that were ultimately developed, the basis of all from a chemical point of view was to chew up and digest some form of natural cellulose, extrude it through a spinneret, solidify it, and draw out a filament which was believed to approach silk in luster and beauty.

It would require courage to say that an industry which now represents approximately one-fifth of all of our textiles is a failure. On a world-wide basis, the great rayon industry of today is, in most senses, an astounding success. In one sense, however, it is a failure. The artificial silks of the early part of this century cannot be compared with the natural product, and it was not wholly surprising that the rayon industry had the good sense to adopt the name "rayon" and desert the concept of an artificial silk. Artificial silk failed to prove an attractive substitute for the expensive and beautiful silk of that period. Its success lay in the fact that rayon, by accident as it were, proved to have qualities that enabled it to replace many articles previously made from cotton, and to develop a volume business that previously had been limited almost entirely to the field of cotton-goods manufacturing.

In a somewhat similar manner, but under vastly different circumstances from a political and economic point of view, we had somewhat the same story in the period between the two great wars when Hitler and Mussolini were attempting to make their countries independent of the rest of the world. Cotton could not be grown in either country, and wool was available only in limited quantities. The production and use of rayon was encouraged and stimulated by the two governments as a substitute for cotton, and the search for artificial wool was greatly encouraged. As a result of this situation, fibers which were remarkable in appearance were developed in both Germany and Italy. They were for the most part protein fibers made from casein or milk. Experiments were tried with other proteins, including soya beans, peanuts, corn, and other products. Unfortunately, from a scientific point of view —although perhaps fortunately from a military point of view—these fibers more closely approximated the wool fiber in appearance than in functional properties. They had somewhat the same hydroscopic characteristics that one finds in wool, which, for certain purposes, make it so attractive. Unfortunately, the dry breaking strength was usually inferior to that of wool, and the wet breaking strength substantially inferior. Due to the cylindrical characteristics of the fiber and the lack of scales, they also lacked that extraordinary quality of wool, unique among fibers, which gives it so many of its desirable properties and which at the same time makes it the

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bane of every housewife from the point of view of shrinkage. These cylindrical, smooth, protein fibers would not full, felt, or shrink. Furthermore, when put to use by the military organization of both countries and tested in the most realistic type of service —namely, in combat—they fell down. I think it is fair to say that valuable as protein fibers in general may be in the future, as blends with other fibers and for specialized uses, it was adequately proved during the war that they fall down sadly when put to the test of serviceability. Again there was failure in the attempt not to improve on nature, but rather to create by mechanical means a product in imitation of nature and at a lower cost.

While all these experiments were being made along the lines of ersatz fibers and fabrics, a great deal of work was being done by scientists in different fields which ultimately lead to discoveries of the greatest importance to the textile industry. Most of us feel that the plastics industry is new. Doubtless the future of the plastics industry is still before it; however, it is also true that plastics is as old an industry as the automobile industry, and as old as others which we now take for granted. The development of celluloid, which might be described as the first of the plastics, dates back beyond the memory of many of us. Celluloid, like the early artificial silks and the protein fibers of the prewar period, was essentially an attempt to substitute a cheap man-made product for an expensive natural one. It was, I believe, an attempt to find a substitute for ivory that lead the chemists of that period to the discovery of celluloid. The important thing about the work that was done with plastics in the early days was the discovery of a phenomenon that is called by the chemists "polymerization." It was found that by treating certain materials with catalysts, or by exposing them to radical changes of temperature, the globular or cluster-like molecules making up the material could be stimulated into reaching out, as it were, for their neighbors and clinging to each other's "coattails," thereby forming long chains of groups of atoms which are known as "linear macromolecules." The science of developing plastics apparently consists of finding appropriate raw materials and methods for rearranging their molecular structure along these lines. It cannot have been a coincidence that someone first noticed that the molecular structure of the raw material of natural fibers is almost invariably made up of these same linear macromolecules. This is true of the keratin from which such natural fibrous materials as hair, hooves, horns, and fingernails are made. It is similar to the structure of cellulose, which is the basis of the cotton fiber. Someone must have recognized this coincidence, and work was started, stemming from the plastics industry, with the goal of producing not a plastic for molding into solid materials, but rather a fiber or filament that could be spun and woven into a textile of a type not known before. This work culminated in the brilliant research activities of Dr. Carruthers of the DuPont Company, and reached its climax with his patents on that great new fiber, nylon, which came out some 12 or 13 years ago.

I wish to emphasize this fundamental difference between the point of view of those who were trying to make an artificial silk and an artificial wool, and the scientists of the DuPont Company and others. I wish to emphasize the difference between the attempt to make an artificial product cheaper than, and perhaps not as good as, a natural product, and the effort to produce a fiber capable of being spun and woven into fabrics bearing no direct relationship to existing products and basing their claims on functional values in an absolute rather than in a comparative sense.

Nylon is, of course, the first of what may properly be called the "new" new fibers. Its abrasion-resistance is outstanding. There is, unfortunately, no accepted index of this value. It can be said, however, that in this respect nylon is probably the best textile fiber known. Its breaking strength of from some 5 to 7½ g./den. makes it the strongest fiber available. Like wool, it has great elasticity and excellent recovery. It has in common with the other high-polymer synthetics that new quality in textile fibers of reacting in certain ways to heat as the more common natural fibers react to moisture—creases and, less fortunately, wrinkles can be set into a nylon fabric by the use of high temperatures, and will remain as constituted until a higher temperature is reached. A remarkable resistance to deformations can be obtained by heat-setting at similar high temperatures. Due to the hydrophobic qualities of the fiber, humidity has little or no effect. Nylon and, to a greater or lesser extent, the other high polymers react to temperature in much the same way as natural fibers do to moisture, and, since in actual service the range of temperatures is vastly smaller than the range of humidity conditions, this quality is of enor-

mous advantage to the new synthetics. Nylon, however, is not that will-o'-the-wisp, the "perfect fiber." Pleasantness of hand and attractiveness of feel is of no consequence in a tow rope or in the folds of a military parachute, but in garments and clothing it is of profound importance, and in this sense nylon falls down. It has a cold and clammy feel which in the continuous-filament form is to most of us quite unattractive. In tricot knit form the hand is improved; fabrics of spun and woven nylon have an even better hand, but manufacturing limitations in the way of warp sizing and spinning of sufficiently even yarns have so far limited the production of such fabrics for commercial use.

I think it is fair to say that when the DuPont Company first introduced nylon for women's stockings, it was extremely lucky in that the silk previously used had become, for obvious reasons, unavailable. The establishment of the nylon stocking was accomplished in a matter of months, whereas ordinarily it might have taken years. Brilliant technical research combined with equally able market research and the fortuitous accident of an international situation made "nylon" a magic name. In that it was blessed, but in another sense it suffered, and I think it deserves our sympathy as well as praise. The magic of the name resulted after the war due to the preposterous situation in which the principles of fabric design and sound garment construction were forgotten and commercial advantage of the shortest term was substituted by the trade. Nylon fabrics of every sort and description were thrown into garment form and offered to the public. Some were good, some were indifferent, and some were disgraceful. Nearly all were over-priced. They did not "breathe," as people said. They were cold and they were clammy, and the seams came apart long before the fabric had even started its useful life. I am firmly convinced that the nylon fabrics and garments marketed in the first years after the war set back the development of new fibers, psychologically at least, most seriously. The public has sometimes blamed the fiber—the industry should blame itself. The future of nylon is, however, assured; but work, and lots of it, remains to be done in order to get the the most from this extraordinary and revolutionary product.

Orlon was first brought out by DuPont as Fiber A. It is an acrylic—*i.e.*, it is made from a polymer of acrylonitrile. It also is not the perfect fiber. It has, however, its own peculiar virtues. Its resistance to acids is extraordinary, and to alkalis is quite good. Not as strong as nylon, it has somewhat the same order of strength as cotton and is much stronger than rayon or wool. It has two outstanding virtues. Its cross section of dumbbell shape gives an irregularity that, combined with a low specific gravity, results in a higher bulking quality than that of any other fiber. In theory at least, therefore, given the appropriate fabric construction and appropriate denier, it should make an ideal fabric where thermal insulation is required. Outerwear and blankets come to mind at once. Its other great virtue is its resistance to sunlight. Where nylon is degraded in varying degrees on exposure to the sun and weather, Orlon is substantially unaffected. The use of this fiber of great resistance to chemical reaction and to sunlight for industrial filtration, and for awnings, ducks, and similar exposed materials is obvious. Furthermore, whereas honesty required comment on the coldness to the touch of nylon, we can honestly boast of Orlon's warm and pleasant hand, which, even if it is against my principles, and for the purpose of comparison only, can be described as "silky." As yet, however, our will-o'-the-wisp escapes us; Orlon has one weakness—it burns. I mention this with trepidation because although the facts are readily determined, their meaning is not so evident. Nylon melts and does not burn; the melt, however, is hot and can result in painfully unpleasant and quite serious damage to the human skin. Molten Orlon continues to flame, and might add to the hazard of a burning fabric. These are facts, but I doubt their significance. In spite of legislation proposed in some areas and on the statute books in others, it seems to me that a society that has got along quite well with rayon and cotton, which are highly combustible, is perhaps too demanding of a new fiber. However, this is a matter more for the consideration of the technologists of the fire-insurance companies than for the textile technologists. Many of us are not wholly in agreement amongst ourselves and, if we cannot evaluate the facts within our family circles, I do not propose and have no hope of finding an exact evaluation in the industry. Orlon curtains are on the market. Orlon underwear is now available. The real and final test of public acceptance will shortly determine this fiber's future in such uses. Orlon's biggest hurdle, I believe, is in the magic named of "nylon." But the public is dis-

criminating, and will, I think, determine what we in the laboratory cannot always foresee.

Acrilan is an acrylic fiber which is notably different from nylon and Orlon in that it is not made by DuPont. Acrilan, made by the Chemstrand Corporation (in which Monsanto Chemical and American Viscose Corporation own equal interests), is made, like Orlon, from a polymer of acrylonitrile. Its characteristics are largely those of Orlon; I will dwell on it no further than to say that while its cross section, which is essentially that of an ellipse with a bite taken out of it, in theory should not result in a fiber with the great bulking power of Orlon, my own observation is that with the same construction of fabric it is hard to judge the difference between them. It is possible that the greatest importance of this fiber lies in the fact that of the half-dozen important new synthetics only Orlon and Acrilan will and should directly compete in the fields of fabric and end-product design. None of the new fibers are perfect. Each has its place, but I believe that Orlon and Acrilon will fill largely the same niche.

Dynel is a partial acrylic made by Union Carbide and Carbon Corporation. It is a most important fiber. In strength it falls below nylon, in resiliency below Dacron, in bulking power a little below Orlon and Acrilon, in chemical resistance it is second to none, and in resistance to combustion it stands alone. Whereas nylon melts and drops its melt below and Orlon and Acrilon have burning residues, dynel not only fails completely to support combustion, but when exposed to flame it merely turns into a hard black char, neither dropping nor melting, and shrivels up into itself. The applications of this fiber are obvious, where chemical resistance is desired and hazards from fire exist. Like the others, it is not the perfect fiber—its weakness, paradoxically enough, is in its vulnerability to heat. Garments made from dynel alone can be ironed on a Hoffman press, but hand ironing is hazardous to the garment. When dynel is blended with viscose rayon or cotton, the cloth, even if dynel is the predominant fiber, appears to change its characteristics, and hand ironing is possible. In addition to its technical qualities, dynel fiber has one important feature. Whereas in staple form the other new synthetics are now offered at prices of from $1.75 to $1.90 per lb., dynel is but $1.25 per lb.* I have little information about the costs of processing or procuring the raw materials used for these high-polymer fibers. Furthermore, I have failed to note any great enthusiasm on the part of the chemical companies for engaging in very specific discussions of their costs. It would seem, however, that vinylchloride is cheap compared to acrylonitrile; and since dynel contains some 60% vinylchloride, it would appear that, no matter what reductions are made in the processing costs of the new synthetics, dynel should retain a competitive advantage with regard to price.

My personal experience with dynel is limited largely to blankets, and in that field I believe history has been made. I like to think that the dynel blanket which does not burn or mildew or shrink, which can be disinfected or cleaned with full-strength household bleaches, which is completely mothproof, and which is no dream of the future, but which has been manufactured successfully and sold in volume, marks a milestone in the march of synthetic fibers along the road started by the nylon stocking.

Dacron is strong, tough, and quite durable; its outstanding characteristic is resiliency. I shall never forget the afternoon when three gentlemen of the DuPont Company called on me and in a provocative way asked me what quality was most needed in a new synthetic. It wasn't hard to hit on resiliency as the lacking feature. Their reply was to throw a crumpled 4-in. sample of cloth into the air and let it bounce back on my desk; it had not a wrinkle, not a crease, and a pleasant hand. That was my introduction to Fiber V or Terylene or Dacron, as it is now named. It is an amazing fiber. Since its cross section is cylindrical, it would not appear to have the bulking power of the acrylic fibers, but the extraordinary qualities of crease-resistance, whether wet or dry, far more than compensate for this in use for garments of all sorts. In continuous-filament form, knitted or woven, its use for women's underwear, blouses, and the like would seem to be assured; in staple form, either by itself or blended with other fibers, it would seem to be ideal for outerwear. The fiber melts, and in this quality would seem to behave somewhat like nylon. My own experience with a Dacron suit has been most satisfactory in all respects except this one. To have thrown the trousers into an automatic washer and then into a hot-air tumbler drier, and then to have put them on and worn them to the office, no iron having touched them, yet with no trace of loss of crease or addition of wrinkle, was an astounding experience. It is true, however, that another example of

* On Jan. 1, 1952, Union Carbide raised the price of dynel to $1.40 per lb. The argument, however, remains unchanged.

our failure to find the perfect fiber is that the dottle from a pipe or hot ember from a cigarette leave scars that cannot be erased. Small holes appear from time to time that cannot be disregarded. I imagine, but have not proved as yet, that blending Dacron with other fibers, perhaps natural ones, will result in fabrics that leave the crease-resistance characteristics of the Dacron undisturbed and, at the same time, reduce the hazard of burns from cigarettes. It is not necessary to dwell on the characteristics of this fiber; its applications are obvious.

There are, of course, other fibers than those mentioned here which could be brought into this discussion. It is my understanding that American Cyanamid Company is in the pilot-plant stage of the development of another acrylic to join the competition between Orlon and Acrilan. Fiberglas has proved itself of great value in certain industrial fields, and has made some advances in the field of curtains and draperies. The protein fibers—Vicara of the Virginia and Carolina Chemical Company, Ardil of the Imperial Chemical Company in England, Merinova of the Snia Viscosa in Italy—in my opinion are of value only because they supply a soft hand in special instances, largely in high-style and fashion work. Their functional characteristics do not put them in the same class as the fibers discussed so far. There are doubtless other fibers made from other materials that will have importance in the future, but I have tried to limit the present discussion to the new fibers of great functional value, for which plans in terms of multimillion pounds have crystalized either to the point of actual production or to the point of bricks and mortar for construction.

What is the impact of these fibers on our industry? It seems to me that those who are engaged essentially in the manufacture and distribution of cotton or rayon fabrics need have little cause for concern. Our production of billions of pounds of cotton and over a billion pounds of rayon is so great that the impact of the new synthetics cannot be of serous concern for many years to come. Exact figures as to production plans for the new high polymers are, of course, not yet readily available; however, plans for the immediate future would not appear to run much above 300 million pounds per annum, and such a figure can be absorbed. The finishers will have to learn more about dyeing and heat-setting. Dyeing problems are being overcome day by day, and heat-setting is essentially a simple process. I think it unlikely that the finishers of silk and rayon will necessarily hold this business to themselves. It would seem that the cotton finishers, who in many respects have had simpler mechanical problems to face than the rayon finishers, will see that the rugged fabrics made from these new synthetics can be handled along mass-production lines and will undoubtedly come into the picture more and more. It is possible that the garment cutters may find some radical changes in their industry. There is no question but that sewing thread will have to be studied with great care; much is yet to be learned in that respect. I think the biggest thing that the garment trade will have to face is the possibility that, because of the characteristics of these new fibers in reacting to temperatures, it may prove desirable to heat-set fabrics after they have been manufactured into garments rather than before. That sounds extremely radical, but, after all, it has been common practise in the manufacture of the nylon stocking; I do not guarantee that this will happen, but I believe that the possibility must be considered. I will not dwell on the effect on the woolen industry except to say that its vulnerability is not merely a matter of the functional values of these new fibers. The volume of woolens produced is not of the order of that of cotton or of rayon, and in that sense the woolen industry cannot so readily absorb the impact of the new synthetics. The woolen industry must watch itself.

The biggest field for exploration, and the field about which those who spin and weave can do the least, is in the design of new machinery. Linen and flax spinning equipment is designed for the long flax staple, woolen and worsted equipment for the woolen staple, and cotton for the cotton staple. Each is designed for the staple length in which raw material has been found in nature. Spun rayon has been with us only for some 15 years, of which at least 5 were busy ones in other fields. Cotton equipment designed for staple lengths of 1 in. to a little more than $1\frac{1}{2}$ in. was readily available, and $1\frac{9}{16}$ in. became the common staple length for rayon. It is evident, however, that the cost of one synthetic staple length is no different from another, and now for the first time the length best suited for the end-product becomes the most appropriate. In the future equipment must be designed and installed to run this length, or, better still, to have such elasticity as to run a relatively wide gamut of lengths. As things stand now, an approximate $1\frac{1}{2}$ in. seems to be the most common

staple length. What it will be in years to come, I do not know, nor have I seen the data necessary on which to base a scientific or orderly opinion; however, instinct, and the guess of others better qualified than I, tells me that it will be 3 in. more or less. I do not know whether the solution lies in direct spinning, in the converter, or in modifications of flax, wool, or cotton spinning; changes there will certainly be, and I am sure that in the years to come the equipment used for the carding and spinning of our new synthetics will differ to an important degree from any of the equipment now commonly installed. Do not misjudge, however, my suggestion. No huge capital expenditures are necessary to run these fibers. All can be handled on the various types of equipment now in common use. Competition, however, will be keen in time, and eventually the question will not be what can be used, but, better still, what should be used. I like to think that this challenge to our machinery manufacturers is, with the cooperation of the industry, already meeting with a ready response.

The challenge to the textile industry itself, however, is even greater. We too must do some work, or ultimately fail. Our industry has been criticized for failure to progress. Not only have our marks been low for accomplishments, but also for effort. I, for one, have always felt that the criticism has been unjust. Dollar comparisons between the percentage of the sales dollar spent on research in the chemical industries and that spent in the textile industry has always seemed to me to be an example of that great crime in logic, the false analogy. The leaders of our industry have not been unimaginative. We led the industrial revolution and paved the way for Detroit and Schenectady with our early ideas of mass production. We made our progress early in the game. It isn't that we fell behind, but that others caught up with us. Had there been evidence during the last generation that research, development, and radical ideas would show a profit, I am sure that those before us would have searched out that dollar with the same initiative with which they searched out other profits. The facts, however, seem to be that while we had, and have, much to learn about the fundamental nature of cellulose, the cotton fiber was a familiar object to the men who handled it. Its behavior and characteristics were largely matters of common knowledge. Furthermore, it was the accepted raw material for a larger industry and there was no other choice.

Similarly, while there were, and are, many things to learn about the more complicated mathematical aspects of fabric geometry, the general nature of a piece of cotton cloth was well established. Free hydrogen, molecules, electrons, and the like, are objects that are familiar only to scientists. A piece of cloth is, and always has been, a household article familiar to all of us. The joy of exploration and its profits dwindle as the unknown areas decline. But now the situation is changed. Instead of nature's raw materials which are familiar to us all, we have a choice of raw materials, some of which are somewhat understood and are either now or soon to be available and some of which are still to come. About these we know very little. We must learn not only how to spin and weave and dye and heat-set, but also how to blend and to get the best from each. Furthermore, in order to design and weave and construct our end-products, we must learn how cloth made from these new products will behave. Is it resilient? Does it need ironing? Will it crease? How should it be laundered? Will it wear? Is it worth the price? All these and other questions must be answered. Most of us believe we know at least some of the answers, but opinions, claims, and thoughts are not enough, and those of us who have the most confidence are those who desire most the confirmation of our beliefs.

It was not in any scientific work that most of us first learned that faith, hope, and charity were the three qualities needed most if we were not to look through a glass darkly. I know that the technologists of the great chemical companies have faith in their products. The textile technologists of the industry have hope for their success. I know that the operating heads of the textile industry will have the enlightened charity to finance and to encourage the research and development programs that we need if we are to get the most in profits and in satisfaction from the opportunities presented by the work of today's organic chemists.

The Role of Synthetic Fibers in the Textile Industry of the Future

L. G. Ray, Jr.*

FOR HUNDREDS of years prior to the early 1900's, natural fibers such as wool, cotton, flax, and silk were the only fibrous raw materials used by the textile industry. Everyone is aware of the rapid growth in the use of viscose and acetate rayons since 1920. In that year, for example, they accounted for less than 1% of the total textile fiber consumed in the United States, while in 1950 improved versions of these same fibers reached the impressive level of 20% of the total fiber consumption. Since the introduction of nylon in 1939, an increasingly large number of synthetic fibers have been prepared to enter the competition for the textile industry's raw material business. I should like to consider here how these new fibers will fit into the textile industry of the future.

Let us regard fibers as the basic raw materials and then ask what the industrial requirements for raw materials are. Three of the more important requirements are availability, price level, and price stability. But the *most* important requirement is that they will allow the manufacture of "wanted" products—products with a high level of functional performance and aesthetic appeal.

The functional performance and, to a lesser extent, the aesthetic appeal of textile products depend upon the properties of the fibers from which they are made. Before we can predict the future of synthetic fibers adequately, we must understand what their properties are, and how they compare with those of the natural fibers.

Each textile fiber can be characterized by a combination of geometrical, mechanical, physical, and chemical properties; these are all important in determining the usefulness of the fiber. I should like to review the properties of some representative synthetic fibers: nylon, Orlon acrylic fiber, Dacron polyester fiber, cellulose acetate, and viscose rayon; to point out how these properties depend upon the chemical and physical structure of the fibers; and to compare the properties of these synthetic fibers with those of wool, silk, and cotton.

All of the properties of a fiber depend, in the final analysis, upon the chemical structure of the polymer from which it is produced, whether the manufacturer be cotton plant, sheep, silkworm, or man. However, the processes by which fibers are manufactured have an important influence on certain properties. We do not know just how nature processes carbohydrates, proteins, air, and water into fibers suitable for textile uses; but all man-made fibers are prepared by a series of basic steps.

The first of these steps is the formation of a massive polymer from which filaments can be formed. In the formation of nylon, for example, the polymer is prepared by condensing a dibasic acid, adipic acid, with a diacid base, hexamethylenediamine. These two chemicals react together: first an acid, than a base, then an acid, etc., until about 100 units are linked together to form a long chainlike molecule. The last stages of this reaction are carried out at high temperatures, where the polymer exists as a viscous melt. The viscosity of molten nylon makes it possible to extrude the material through small holes in a spinneret plate to form filaments, as shown in Figure 1. These filaments solidify in the air below the spinneret and are collected on a suitable package. Nylon

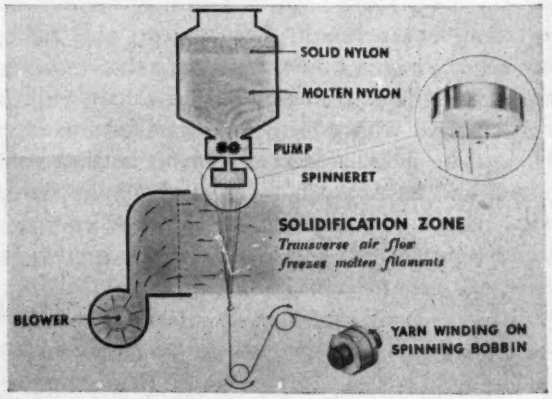

FIG. 1. *Melt-spinning nylon.*

* Technical Division, Textile Fibers Department, E. I. du Pont de Nemours and Co., Inc., Wilmington, Del.

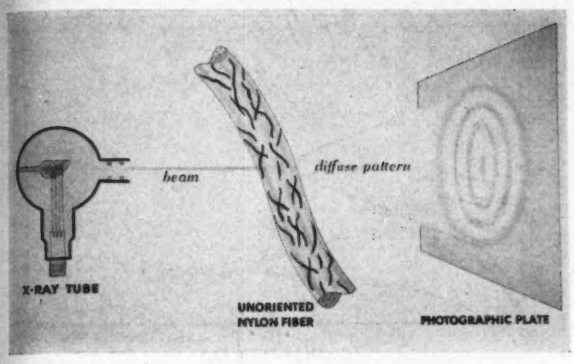

FIG. 2. *X-ray diffraction pattern of unoriented nylon fiber.*

filaments at this stage of manufacture are weak and stretchy, and are of little value for general textile use, because the individual nylon molecules are coiled up and randomly oriented in the filaments. We can illustrate this by reference to the x-ray diagram shown in Figure 2. X-rays pass through the fiber, are diffracted by the molecules, and produce hazy circles on a photographic plate mounted behind the fiber. This type of diffraction pattern shows that the molecules in the fiber are completely unoriented.

In order to develop useful fiber properties, individual polymer molecules must be aligned. This alignment is accomplished by stretching the unoriented fiber, as indicated in Figure 3. During the stretching operation, the fiber necks down and is permanently extended, becoming strong and elastic. The change which takes place within the fiber can be demonstrated again by reference to an x-ray diffraction pattern (Figure 4). Instead of hazy circles, we now observe a clean-cut pattern of sharp spots and arcs on the x-ray diagram. This indicates that as the fiber draws, the long molecules untangle as they slide past one another. The internal friction produced uncoils the chains and aligns them in the direction of the fiber axis. In many regions, these extended chains pack together in a crystalline lattice,

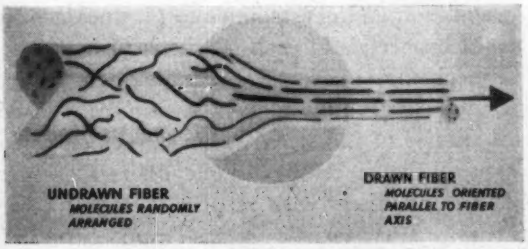

FIG. 3. *Alignment of nylon molecules by stretching fiber.*

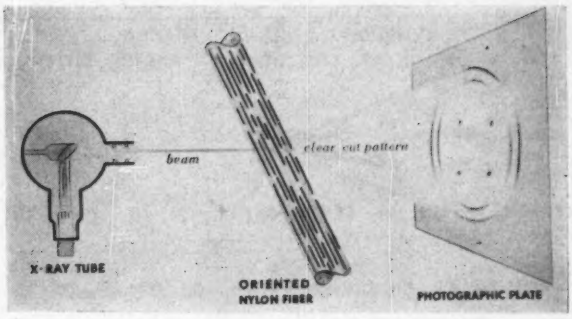

FIG. 4. *X-ray diffraction pattern of oriented and crystallized nylon fiber.*

but the x-ray pattern shows that the long chains never completely untangle. In some areas, coiled chains remain interspersed between the crystallites; these areas are called "amorphous."

All of the mechanical properties of fibers are highly dependent upon the orientation and crystallization of the molecules. The degree of orientation and crystallinity can be controlled in the process of fiber manufacture. From a single polymer it is thus possible to prepare a family of fibers which differ among themselves in mechanical properties all the way from ones which are weak and limp to those which are strong and stiff. However, the range of fiber properties obtainable from a single polymer is always limited, and it does not appear that any one polymer can meet all the requirements of the textile industry. Polymers with new chemical structures are continually being synthesized in an effort to prepare fibers with new and unique combinations of properties. There are at least three elements in chemical structure which are important in determining fiber properties. These elements are: the flexibility of the molecular chains; the number, kind, and distribution of the attractive forces between chains; and the nature of other groups along the chains. These factors can be illustrated with the structures of several synthetic fibers. A knowledge of these structures will be helpful later in explaining fiber properties.

The nylon molecule is a flexible chain of carbon atoms with amide groups interspersed along it at relatively wide intervals. The amide groups of adjacent chains are strongly attracted to one another by hydrogen bonds, as shown diagramatically in Figure 5. The molecules in Orlon acrylic fiber likewise are flexible chains of carbon atoms, but in this case CN groups are attached to every second carbon in the chain. As Figure 6 illustrates, the polar CN groups are bound to CH groups in adjacent polymer mole-

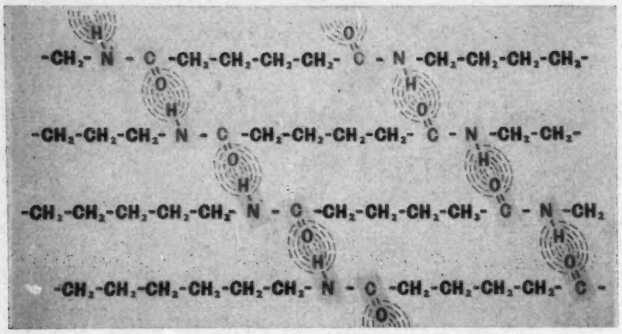

FIG. 5. *Crystalline lattice of nylon.*

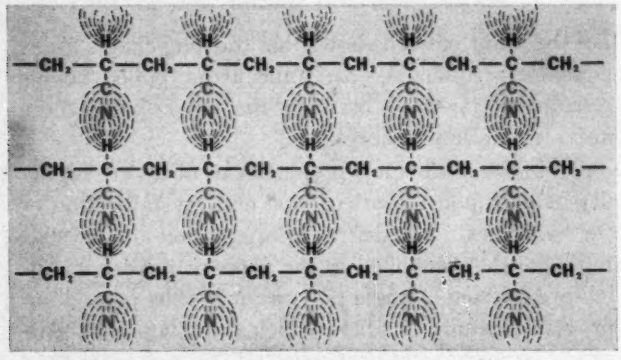

FIG. 6. *Crystalline lattice of Orlon acrylic fiber.*

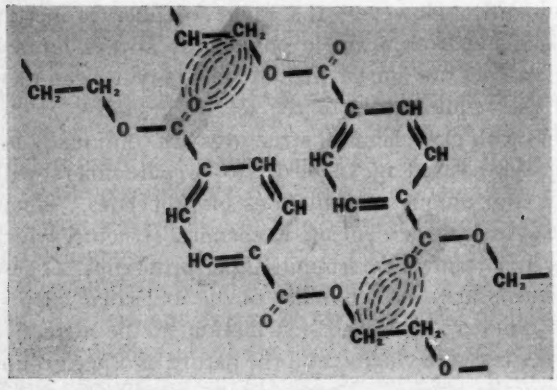

FIG. 7. *Crystalline lattice of Dacron polyester fiber.*

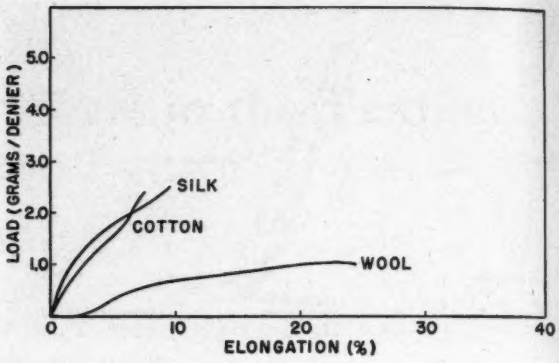

FIG. 8. *Stress-strain curves for natural fibers.*

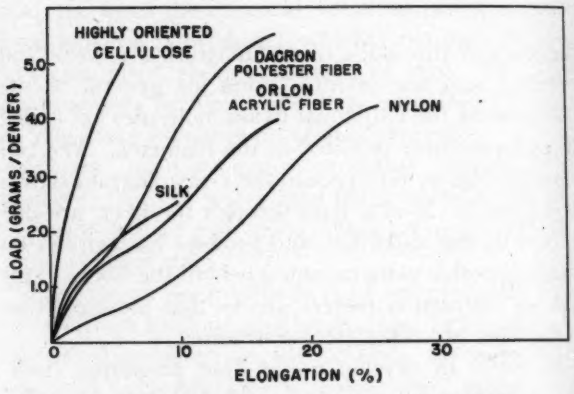

FIG. 9. *Stress-strain curves for "silklike" fibers.*

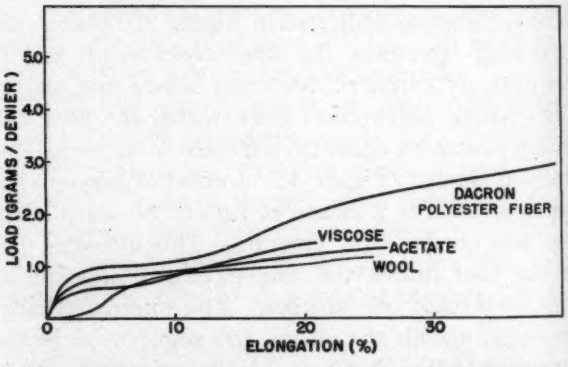

FIG. 10. *Stress-strain curves for "wool-like" fibers.*

cules. While each bonding force is not as high as that between amide groups, there are so many points of bonding that the total attractive force between molecules in Orlon is greater than that in nylon. The molecules in Dacron polyester fiber are inherently stiffer than the two previous examples because they contain rigid benzene rings. The forces between each polyester group (Figure 7) are intermediate in strength between those of nylon and Orlon.

To summarize, elements of chemical structure such as intermolecular bonding and molecular flexibility can be combined with elements of physical structure like orientation and crystallinity to produce a different set of properties in each synthetic fiber.

The mechanical properties of textile fibers are most important in determining their usefulness. Fundamental studies of the stress-strain curves of various fibers have shown that synthetic fibers closely resemble natural fibers with respect to some of these properties. Charcteristic stress-strain curves for wool, silk, and cotton are shown in Figure 8. In Figure 9 are reproduced a number of stress-strain curves

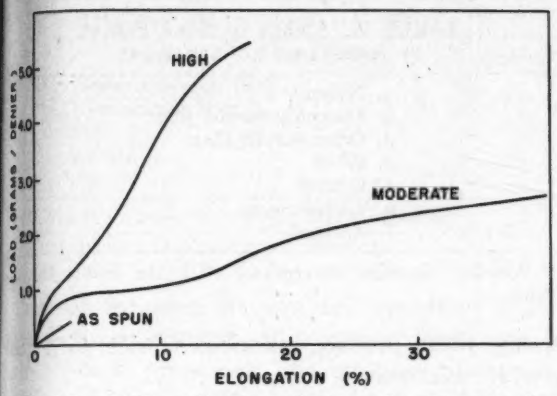

FIG. 11. *Effect of orientation on the stress-strain properties of Dacron polyester fiber.*

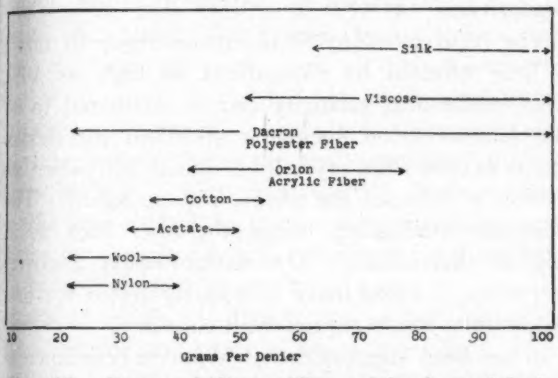

FIG. 12. *Range of fiber stiffness (elastic modulus).*

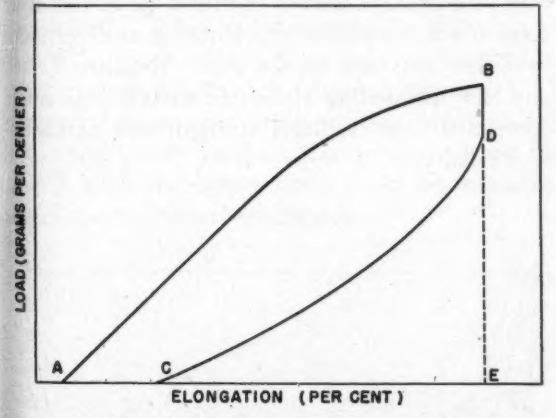

FIG. 13. *A cycle of extension and return.*

which resemble that of silk; and in Figure 10 a number of curves which resemble that of wool. It is significant that Dacron appears in both of the last two figures; this is due to the fact that the stress-strain curve of Dacron, like that of many other synthetic fibers, can be highly modified by differences in the degree of orientation imparted to the fiber during manufacture. This is illustrated in Figure 11, where the stress-strain curves of Dacron having different

TABLE I. TENACITY-ELONGATION RANGES

	Tenacity (g./den.)	Elongation (%)
Nylon (regular)	4.5–5.0	25–18
Nylon (high-tenacity)	6.5–7.7	20–14
Nylon (staple)	3.8–4.5	37–25
Dacron polyester fiber (5600)	4.4–5.0	22–18
Dacron polyester fiber (5400)	3.0–3.9	40–25
Orlon acrylic fiber (type 81)	4.7–5.2	17–15
Orlon acrylic fiber (type 41)	2.0–2.5	45–20
Viscose (regular)	1.5–2.4	30–15
Viscose (high-tenacity)	2.4–4.6	20–9
Acele acetate rayon	1.3–1.5	30–23
Wool	1.0–1.7	35–25
Cotton	2.0–5.0	7–3
Silk	2.2–4.6	25–10

degrees of orientation are compared. These curves were all obtained on fibers which had previously been boiled-off in water.

The fact that orientation and crystallinity can be controlled means that the range of mechanical properties such as tensile strength and elastic modulus of synthetic fibers is much broader than the ranges characteristic of natural fibers. Table I shows the tensile strengths and elongations of several fibers that are available either for sale or for fairly large-scale market development work. Practically all of the synthetic fibers *can be made experimentally* stronger or weaker than the values listed. The relatively high strengths attainable with synthetic fibers like nylon, Orlon, Dacron, and viscose rayon are possible because of the high molecular orientation which can be obtained with these fibers and because of the strong forces existing between the molecular chains. Acetate fibers are generally weaker than cellulosic fibers because the acetyl groups cover hydroxyl groups which in cellulose hold the chains together by strong hydrogen bonding. The low tensile strength of wool is probably due to the fact that the fiber is only slightly oriented, and its chains fit together too imperfectly as a result of their bulky side-groups to allow the development of strong forces between them.

The ranges of stiffness (elastic modulus) available in fibers are shown in Figure 12. Those fibers having the widest ranges are characterized by strong forces between their molecular chains and by the fact that their orientation can vary between fairly wide limits. The low stiffness of nylon is no doubt due to the inherent flexibility of its molecular chains and the relatively wide spacings between its strong bonding groups.

Another important mechanical property of textile fibers is their recovery from deformation. This is

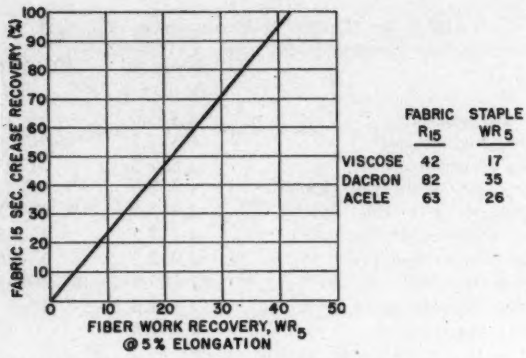

FIG. 14. *Fiber work recovery* vs. *fabric crease recovery.*

usually evaluated by cyclic stress-strain tests like that illustrated in Figure 13. The results of such tests can be described in two ways: by length recovery or by work recovery. Work recovery is the ratio of the area underneath the recovery curve (CD) to that under the extension curve (AB). The work recovery of fibers has been related to the crease recovery of fabrics, as shown in Figure 14. This correlation is quite good for certain fibers, like those indicated, but breaks down for others, such as nylon and polyethylene, where crease recovery is generally lower than would be expected from the work recovery values.

The work recovery of fibers from elongations of 1%, 3%, 5%, and 10% is shown in Figure 15. Dacron polyester fibers have recovery properties very similar to those of wool, and these fibers produce the most resilient fabrics. The recovery properties of acetate rayon are similar to those of wool and Dacron; their stress-strain curves are also alike.

TABLE II. ORDER OF SOME FIBERS IN RESISTANCE TO ABRASION*

1. Nylon
2. Dacron polyester fiber
3. Orlon acrylic fiber
4. Wool
5. Cotton
6. Viscose rayon
7. Acetate

* Wet-flex abrasion determined with the Stoll Abrasion Tester.

Acetate fibers produce luxurious fabrics with good wrinkle-resistance.

The recovery properties of Orlon and silk are very similar, and fabrics from these two fibers reflect this similarity.

The recovery of nylon is extraordinary in that it is little affected by elongations as high as 10%. This outstanding elasticity can be attributed to the fact that as nylon fibers are stretched the flexible chains between the crystallites uncoil, but when the tension is released the chains coil up again. This molecular mechanism brings the fiber back to its original dimensions. The extraordinary recovery properties of nylon make it uniquely suited for uses like hosiery which require high elasticity.

It has been suggested that abrasion-resistance of fabrics is related to the strength and to the recovery properties of fibers. Table II gives the order of some fibers in resistance to abrasion as determined by wet-flex tests run on the Stoll Abrasion Tester. Nylon has outstanding abrasion-resistance, as might be predicted from its high strength and extraordinary elasticity.

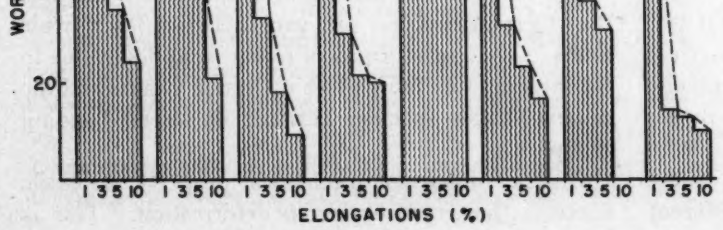

FIG. 15. *Fiber work recovery from various elongations.*

TABLE III. CREASE RETENTION

Fiber	% Crease retained*
Dacron polyester fiber	85
Orlon acrylic fiber	85
Wool	20
Acetate	15
Viscose	5

*$\frac{\text{Height of crease after wetting fabric in water}}{\text{Height of crease before wetting}} \times 100.$

Perhaps the most striking and important differences between natural and synthetic fibers are in their physical properties. In moisture absorption, for example, some of the newer synthetic fibers are in an entirely different range from wool, silk, and cotton; this is illustrated by the regain curves in Figure 16. Nylon, Orlon, and Dacron absorb very small amounts of water even at high humidities. This is a result of the lack of large numbers of strongly water-absorbing groups in their molecular structure and of the perfection of packing of their chains leading to high crystallinity. Acetate rayon absorbs less water than viscose rayon because acetyl groups have replaced large numbers of the strongly absorbing hydroxyl groups in cellulose.

Lack of water absorbency has several important effects on the properties of fibers and fabrics. For example, relative humidity affects the mechanical properties of nylon, Orlon, and Dacron to only a small extent. This is illustrated by the data in Table III, which shows the effect of wetting on creases ironed into fabrics from various fibers. It is apparent that even after wetting, fabrics from Dacron and Orlon maintain their creases almost unchanged, while the creases in wool, viscose rayon, and acetate rayon essentially disappear.

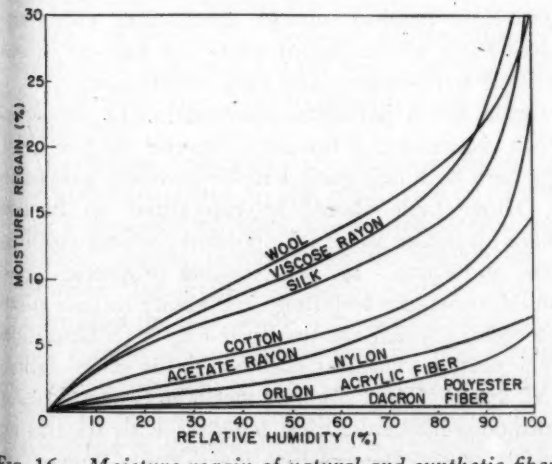

FIG. 16. *Moisture regain of natural and synthetic fibers.*

TABLE IV. HEAT SENSITIVITY OF FIBERS

	Melt (°C)	Stick (°C)	Decompose (°C)	Char (°C)
Nylon	250	235	—	—
Dacron	250	235	—	—
Orlon	—	235	—	—
Acetate	230	200	95–105	—
Viscose	—	—	180	180
Cotton	—	—	150	—
Wool	—	—	135	300
Silk	—	—	150	—

The consequences of low moisture absorption are not all good, however. In general, fibers which do not absorb moisture are more difficult to dye than those which are moisture-sensitive, and are also more prone to develop objectionable static during processing or during wear. Both of these problems are major concerns to synthetic fiber producers, and progress is being made toward solving them.

The effect of heat on fibers is an important practical consideration. Here again fibers differ markedly in their reactions to high temperatures. This is illustrated in Table IV, where data are presented on melting point, sticking point, decomposition temperature, and charring temperature. These data have been collected from various sources [1, 2], and are probably not strictly comparable. However, they serve to illustrate several important points. Nylon, Dacron, and acetate fibers are thermoplastic in the sense that they soften and finally melt at elevated temperatures. Orlon acrylic fiber has no very definite melting point, although it softens and decomposes at high temperatures. Some of the other fibers listed do not soften and melt, but they react in less obvious ways to high temperatures—they decompose at a measurable rate, or char at temperatures which in many cases are below the sticking temperatures of the thermoplastic fibers. The thermoplastic nature of nylon, Orlon, and Dacron has some advantages to offset the obvious but not too serious danger of rapid destruction by heat. It makes it possible to heat-set such fabrics to shape, to put in creases which are permanent even to wetting out in water, and to overcome by high-temperature dyeing procedures some of the dyeing difficulties resulting from low moisture absorption. Incidentally, the heat sensitivity of Orlon, Dacron, and nylon need not interfere with proper tailoring or pressing. Orlon has the best ironing properties of any synthetic fiber in that wrinkles are easily removed and creases ironed in and ironed out with no difficulty. The Orlon fiber is held together by many rather

TABLE V. RELATIVE BULK OF A SET OF COMPARABLE FABRICS

Fabric from:	
Orlon acrylic fiber	130*
Dacron polyester fiber	100
Nylon	100
Wool	100
Viscose/acetate blend	90

* Based on wool as 100.

weak bonds which easily thaw out with the heat of the iron. This is less true of Dacron, and not at all true of nylon, where the bonds cannot be thawed out by safe ironing temperatures.

Bulk is one important physical property of fabrics which has not been related satisfactorily to fiber properties. In the practical sense, bulk means high fabric thickness and covering power at low fabric weight. It is believed that fiber density and fiber cross-sectional shape are two factors which control the bulking potential of various fibers, but other factors such as crimp and frictional characteristics must also be important since these two do not account for all of the differences which have been observed. Data on the relative bulkiness of a set of comparable staple fabrics are shown in Table V.

We believe that the relative bulkiness depends upon fabric construction as well as upon fiber content. In this particular comparison Orlon is outstanding in bulk. Since fabric warmth is generally associated with fabric thickness, the conclusion is indicated that Orlon can be used to produce warm but exceptionally lightweight woven fabrics. Quite aside from the aesthetic appeal of such fabrics, important savings in fiber costs might result from the production of more yards from less weight.

I shall not discuss the chemical properties of fibers in any detail, for this subject is too extensive and I know of no way to select the items of most general interest. It is pertinent, however, that Orlon is highly resistant to chemical attack, presumably because of its highly organized and impervious structure. A comparison of the outdoor durability of several fibers is shown in Figure 17. Orlon is markedly superior to the other fibers in this test. Curves for Dacron and nylon would probably fall some where between those for cotton and Orlon.

I have attempted in this presentation to consider fibers as raw materials for the textile industry. The role of any fiber in the industry of the future will be determined by what it can contribute in functionality *versus* what it costs. The newer synthetic

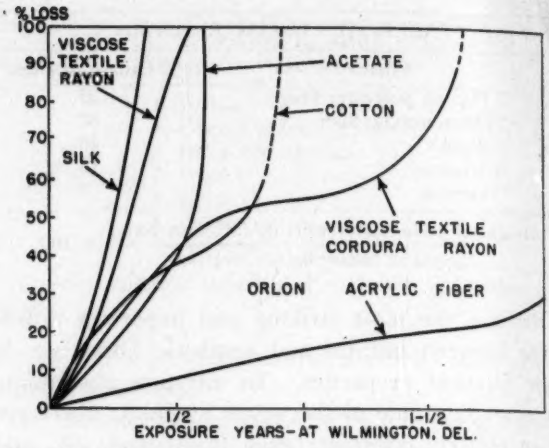

FIG. 17. *Effect of outdoor exposure on yarn tenacity.*

fibers are relatively expensive, and are likely to remain so for some time. This means that their use must be in fields where they can make a real contribution.

By way of illustration, let us consider some of the synthetic fibers discussed herein. Each fiber has some property or combination of properties which is unique. Each fiber should be used alone or in combination with other fibers where its unique characteristics give it the best chance to contribute to fabric performance.

Nyon has outstanding strength, elasticity, and abrasion-resistance. It should be used in fabrics where these properties are important. Stockings, truck and airplane tires, rugs, transportation upholstery, and sports uniforms are recommended uses for nylon.

The most outstanding property of Dacron is wrinkle-resistance, even when wet. Sheer dress and blouse fabrics from continuous-filament yarns and lightweight tropical suitings from staple yarns represent fields where this property can pay off in outstanding performance and easy maintenance.

Orlon has a particular combination of properties which recommend it for use in several specific fields. The high bulk and good wrinkle-recovery properties of Orlon staple should be capitalized on in uses where insulating value is important—winter clothing, suits, and coats. Its easy ironing properties, good handle, resistance to pilling, and ability to take pleats and creases which are unaffected by high humidities are important characteristics for highly styled fabrics of all kinds. The "silklike" mechanical properties of continuous-filament Orlon together with its ironing properties and its moisture insensitivity suggest its

possible use in luxury fabrics which are highly functional. Finally, the outdoor durability of Orlon makes it particularly suited for uses requiring all-weather exposure.

This discussion would be incomplete if it were not pointed out that the textile fibers now in existence may be only the first of many new fibers and may themselves be improved. We are confident that once the characteristics desired in a new textile fiber are clearly enough defined, research has a good chance to produce a fiber with those characteristics. One of the major concerns of the textile industry should be to define more completely what properties are wanted in new textile raw materials.

The synthetic fibers discussed here and others which are in various stages of manufacture or development have properties which make them valuable additions to the textile industry's list of basic raw materials. *But they are only raw materials.* Their future depends upon the skill and ingenuity which can be brought to bear in designing consumer products which will take advantage of their unique properties. This job is a challenge to the synthetic fiber industry and to the textile industry, but it is also an *opportunity* unparalleled in the long history of textile development. The new degree of freedom offered the textile technologist, the designer, and the converter by synthetic fibers opens the way to fabrics and garments which can set new standards in style, performance, and easy care.

New raw materials and new technology for using them will eventually pay off for any industry in improved products—they always have and they always will.

Acknowledgment

The data included in this presentation resulted from the work of many individuals in the Textile Fibers Department of E. I. du Pont de Nemours and Co., Inc. Their contributions are sincerely appreciated. Particular mention should be made of Drs. C. E. Black, J. T. Rivers, and J. W. Ballou, who cooperated in the writing of major sections of this manuscript.

Literature Cited

1. Newell, W. A., Synthetic Fiber Table, *Textile World* **101**, No. 9, 105 (1951).
2. Hartsuch, B. E., "Introduction to Textile Chemistry," New York, John Wiley & Sons, Inc., 1950, p. 122.

Book Reviews

Textile Inter-Fiber Competition 1937 & 1949. Supplement to *Rayon Organon.* New York, Textile Economics Bureau, Inc., 1951. 16 pages. Price, $1.00.

This report presents the results of an exhaustive survey of textile fiber end-use consumption for the year 1949, and compares the data with those obtained in a similar study conducted 12 years ago for the year 1937.

Composite tabulations are shown of the poundage of fibers consumed in the production of men's and boys' apparel; women's, misses', children's, and infants' apparel; products for household uses; and products for industrial uses. Cotton, wool, silk, linen, and the man-made fibers are covered. A striking comparison of the over-all end-use changes in fiber consumption is shown in one of the tabulations. This over-all data reveals the penetration of all major divisions of the textile industry by rayon and other man-made fibers.

Review of Textile Progress: Vol. I: 1949. Manchester, England, The Textile Institute, 1950. 403 pages. Price, $3.25.

This is the first issue of what is projected to appear as comprehensive annual reviews of current progress in textiles. The book is classified into 10 principal divisions, following somewhat the pattern of the Abstracts Section of the *Journal of the Textile Institute.* Each division is subdivided to include the principal fields of activity, and each subject is discussed by a well-known British authority in the field.

The general pattern is that of an annotated bibliography with, however, the references cited appearing at the bottom of each page. The obvious disadvantage to the informed researcher due to this arrangement is overcome to a considerable extent by the inclusion of author and subject indexes at the end of the volume.

A large amount of information concerning the subject matter of the literature cited has been condensed

into this review, and it should prove to be a useful reference to all persons engaged in the production of fibers, their conversion into end-use products, and/or research.

Cotton from Field to Fabric. Prepared by the Staff of the National Cotton Council. Fifth Edition. Memphis, Tenn., National Cotton Council, 1951. 52 pages. Price, $3.25.

This edition presents an up-to-date and extensive revision, reorganization, and enlargement of the first edition, published in 1939 as *Cotton from Raw Material to Finished Fabric.*

Following a brief historical discussion of the growth of the cotton industry, concise descriptions are given of the techniques of cotton culture and harvesting, manufacturing processes, quality characteristics, and peacetime and military uses. Of special importance is the section in which are displayed 112 sample swatches of cotton fabrics representing standard constructions, with several pages devoted to definitions and descriptions of the principal classes of fabrics.

A wealth of information is condensed into the book. It will be of interest to all persons who are in any way connected with the cotton industry, including textile merchants and retailers, and to schools.

Know Your Fabrics. Lucy D. Taylor. New York, John Wiley & Sons, Inc., 1951. 366 pages. Price, $6.75.

In the sense that scientists and technologists are likely to become concerned with problems connected with the selection of appropriate textile furnishings for their homes, they are decorators. *Know Your Fabrics* has to do with decorative textiles. The subject matter is based on the broad experience, as teacher and decorator, of Lucy D. Taylor, now Lecturer, New York School of Interior Design.

Miss Taylor has used excellent judgment in her selection of material and her arrangement of it. In Section I are discussed the principal standard fabrics, what they are, and how they may be used most effectively. Section II, "The Characteristic Expressions of Historic Fabrics," deals with the historical background and development of fabrics during the Early, High, and Late Renaissance periods in Italy; during the English, French, and Spanish Renaissance; and in France from 1643 to 1815. From this section one can gain an appreciation and understanding of the possibilities for employing items of widely varying source and still avoid dull or bizarre effects. The final section is entitled "Choosing Coverings for Chairs of Today." The excellent illustrations of fabrics as such and of fabrics in place on furniture contribute to an understanding of the principles involved in the selection of furnishings, as well as contributing to the attractiveness of the book.

Well written and concise, with a wealth of practical information and a moderate price, *Know Your Fabrics* should not only be of great assistance to students, but should also be a valuable addition to the home bookshelf.

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